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Castings and Processing of Uranium and Uranium Alloys

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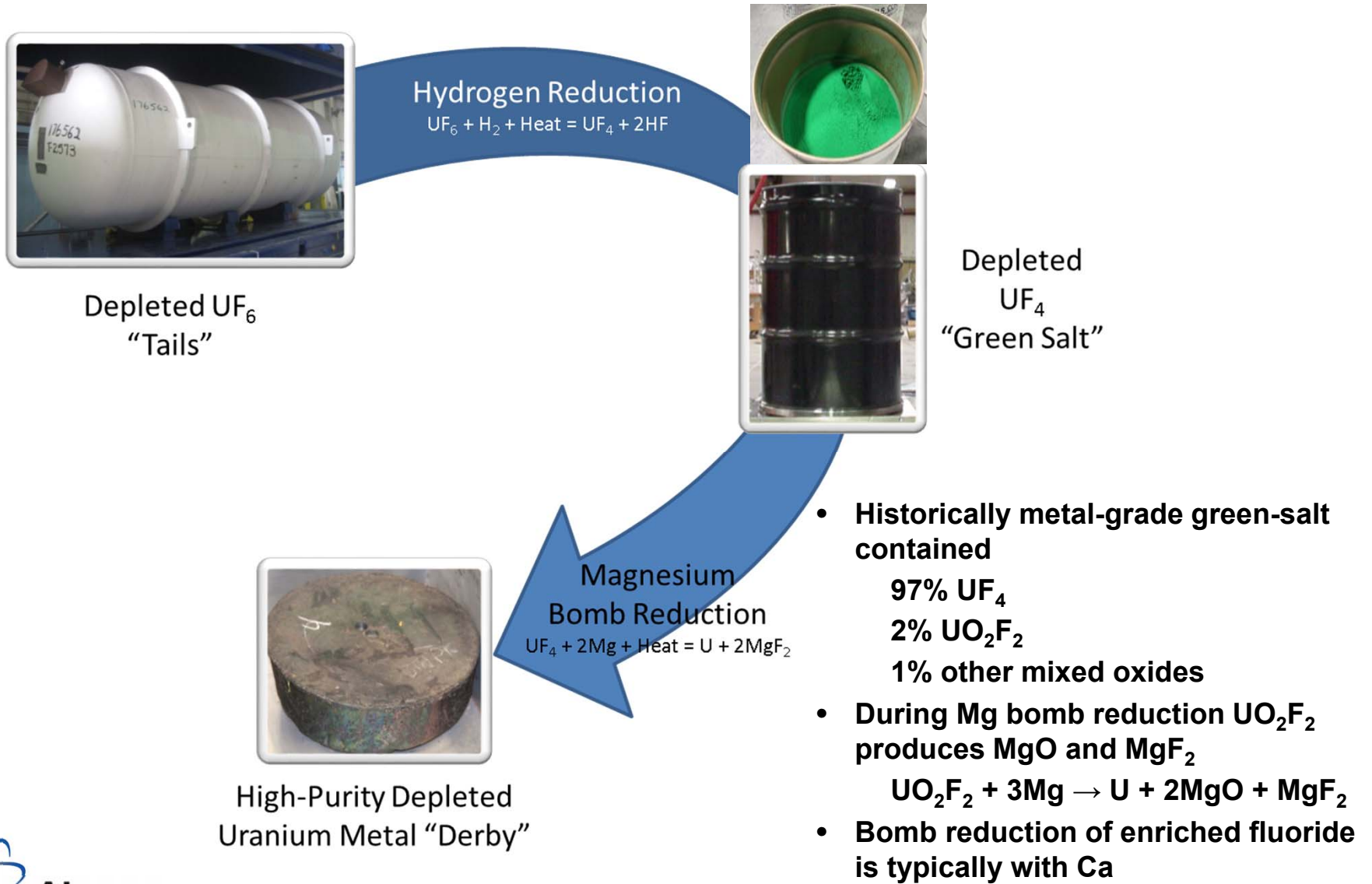
Outline

- **Production of DU Derby**
- **DU Casting and Processing**
 - Vacuum Induction Melting (VIM)
 - Cast DU Microstructure / U-C(-N) Phase Diagram
 - Wrought DU
 - DU Hydro Plate and DU Specifications
- **Enriched Uranium**
- **Wrought U-6Nb**
 - Vacuum Arc-Remelting (VAR)
 - U-6Nb Ingot Production (and Variations)
 - Wrought Processing
 - U-Nb Macrostructure
- **Direct Cast U-6Nb**
- **Plasma Arc Melting (PAM)**

Processing of Unalloyed Depleted Uranium

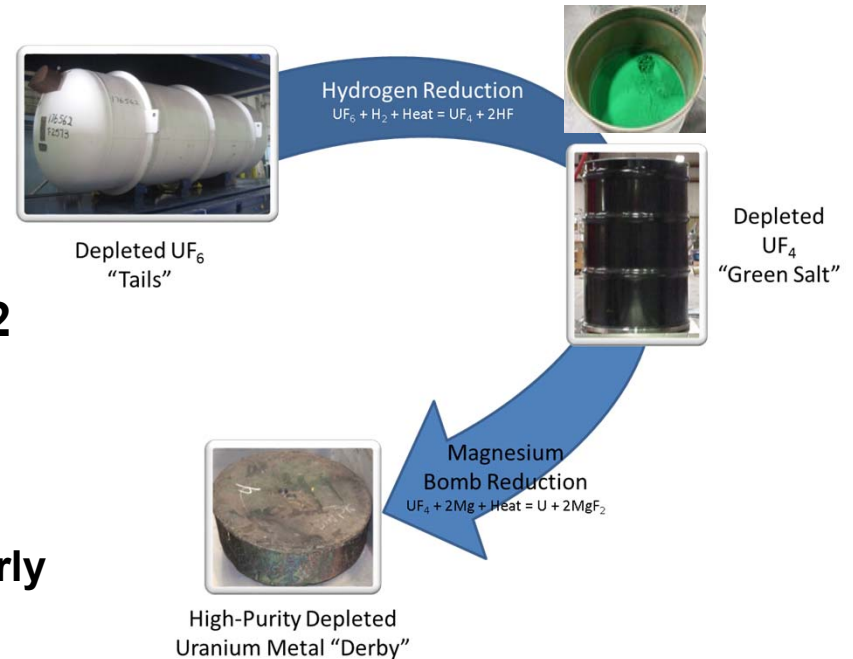
- **Production of DU Derby**
- **DU Casting and Processing**
 - **Vacuum Induction Melting (VIM)**
 - **Cast DU Microstructure / U-C(-N) Phase Diagram**
 - **Wrought DU**
 - **DU Hydro Plate and Specifications**

Production of DU Derby



DU Derby / High Purity DU (HPDU)

- Fernald Plant was the sole source supplier for HPDU prior to its shut down in 1990's
- Starmet did UF_6 to UF_4 to derby conversion for Y-12 from 1999 to 2002
- Since 2002
 - Aerojet has been qualified to convert existing inventories of UF_4 into derby
 - UF_4 that meets WR specification is nearly exhausted
 - No US capability in to convert UF_6 to UF_4
- Paducah and Portsmouth sites are currently storing 1155 cylinders of UF_6 that meet WR requirements (~9 ton DU metal each cylinder)
- In 2015, Y-12 issued an EOI for a commercial vendor to provide conversion of (1) UF_6 to UF_4 and (2) UF_4 to DU metal.
 - 6,800 MTs of DU for future DOE/NNSA mission deliverables
 - Response came it at \$25M to stand up UF_6 to UF_4 plant and \$85/kg of metal
 - Plan is currently on hold



Vacuum Induction Melting

- Vacuum induction melting (VIM) utilizes induction melting to melt metal within a vacuum
- In induction melting an alternating magnetic field is produced by a coil being driven by an AC power supply
 - Typical VIM frequencies are 1 to 10 kHz
 - As furnace becomes larger, typically the frequency is lower
- The alternating magnetic field induces eddy currents in the conductive metal/mold/crucible within the coil and the conductive material heats resistively
- Coil is typically water cooled
- Insulation between mold stack and coil prevent heating of the coil
 - Non-electrically conductive insulation is not heated by the alternating magnetic field



36" x 18" ϕ Split Coil

Vacuum Induction Melting of Uranium (1)

- Use graphite molds and crucible
 - Relatively inexpensive to produce
 - Good high temperature properties and thermal shock resistance
- Use ceramic mold coating to avoid chemical reaction between uranium and graphite
 - A variety of oxide coatings work
 - Historically coated with ZrO_2
 - Use yttria Y_2O_3 (DU) or erbia Er_2O_3 (EU)
 - DU generally use yttria with a cellulose binder and water or alcohol carrier
 - Must be be mindful of binder burn-out temperature; generally mold must be above 750°C or can get gas porosity
- Rules of thumb for cast shapes
 - Bottom of mold at about 800°C
 - Top of mold at about 1100°C
 - Metal at 1300 to 1325°C (for unalloyed)
 - If charge contains derby, hold molten for 45 minutes to diminish bomb reduction products

Yttria Coated Mold



Erbia Powder

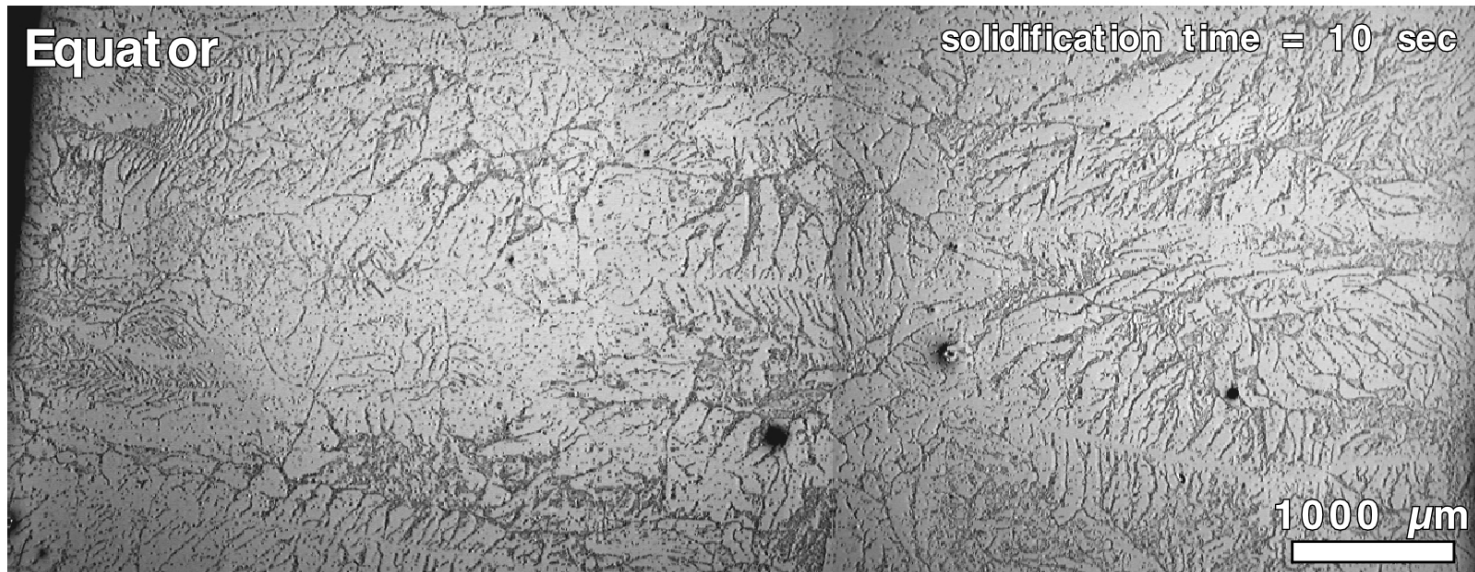
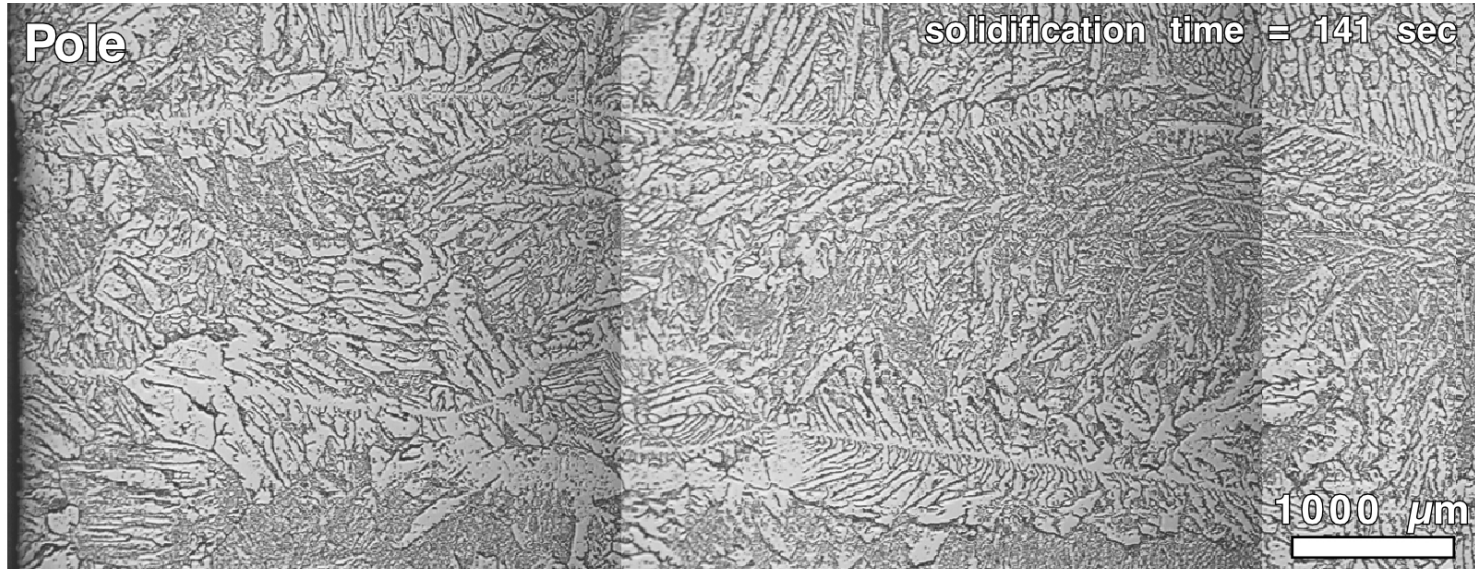


Vacuum Induction Melting of Uranium (2)

- **Generally pick up carbon each time uranium is melted**
 - Rule of thumb: get increase of ~ 25 ppm wt each time uranium is melted
 - Pick up is though gas phase transport
 - O_2 in furnace atmosphere reacts with graphite to form CO
 - CO reacts with molten metal surface to form UO_2 and carbon in solution
 - Imperfect mold coating may contribute to surface layer
 - High vacuum leak rate, higher casting temperatures, and long hold times all contribute to increased carbon pickup
- **Casting surfaces generally have a rough ~0.050" thick oxide layer on outer surface that must be machined off**
- **Generally use bottom pour crucible to avoid entrainment of crucible surface oxide into the cast part**
 - Both stopper rod and rupture disks are common
 - Oxide remaining in the crucible after pouring is called the *skull*
- **With melting and casting, the radiologic decay (daughter) products ^{234}Th (24 day half-life) and ^{234}Pr become concentrated in the crucible skull and casting hot top**
 - Readings on skull may be as high as 5 R beta on contact

Cast DU Microstructure (1)

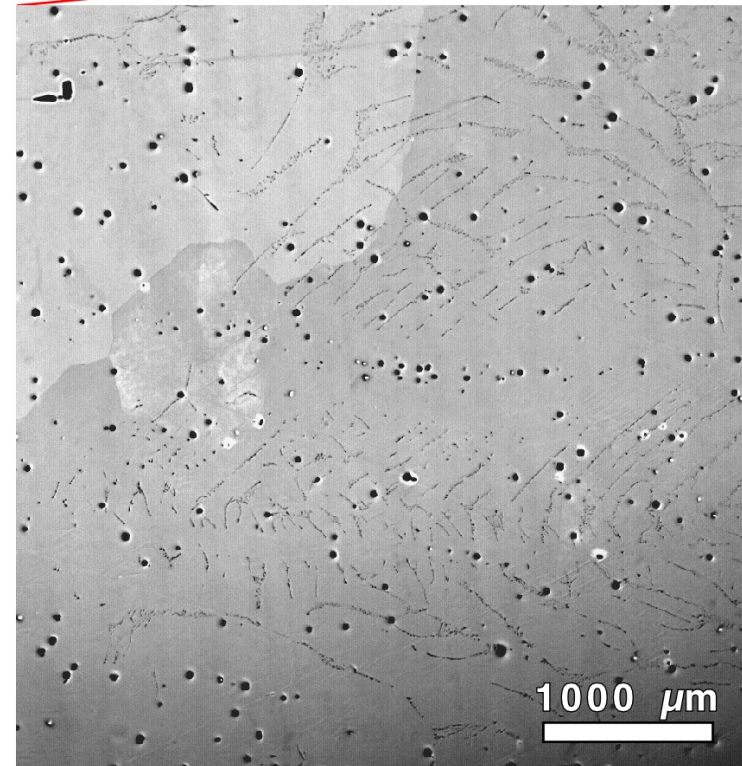
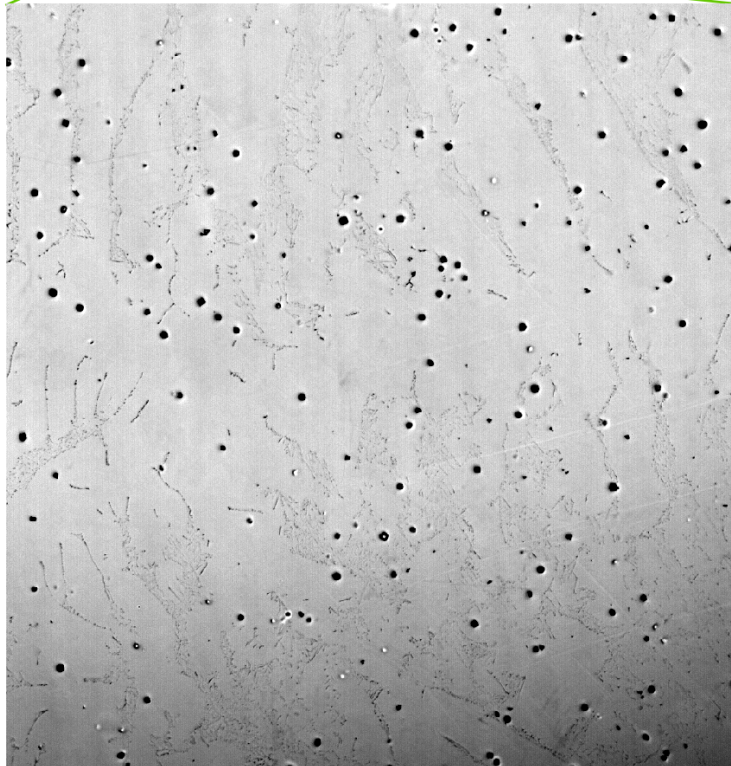
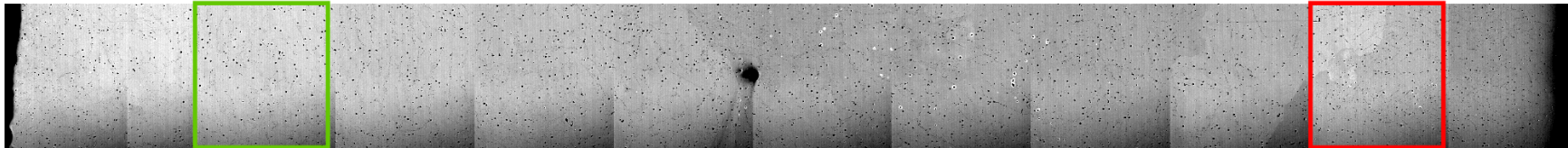
04K-448 Basic 150 mm Hemi



Optical micrographs of as electro-polished samples
(27% phosphoric acid - 27% ethylene glycol - ethanol)

Cast DU Microstructure (1)

Metallography of 04K-448 at Equator
Image across the full 1 cm of the casting thickness

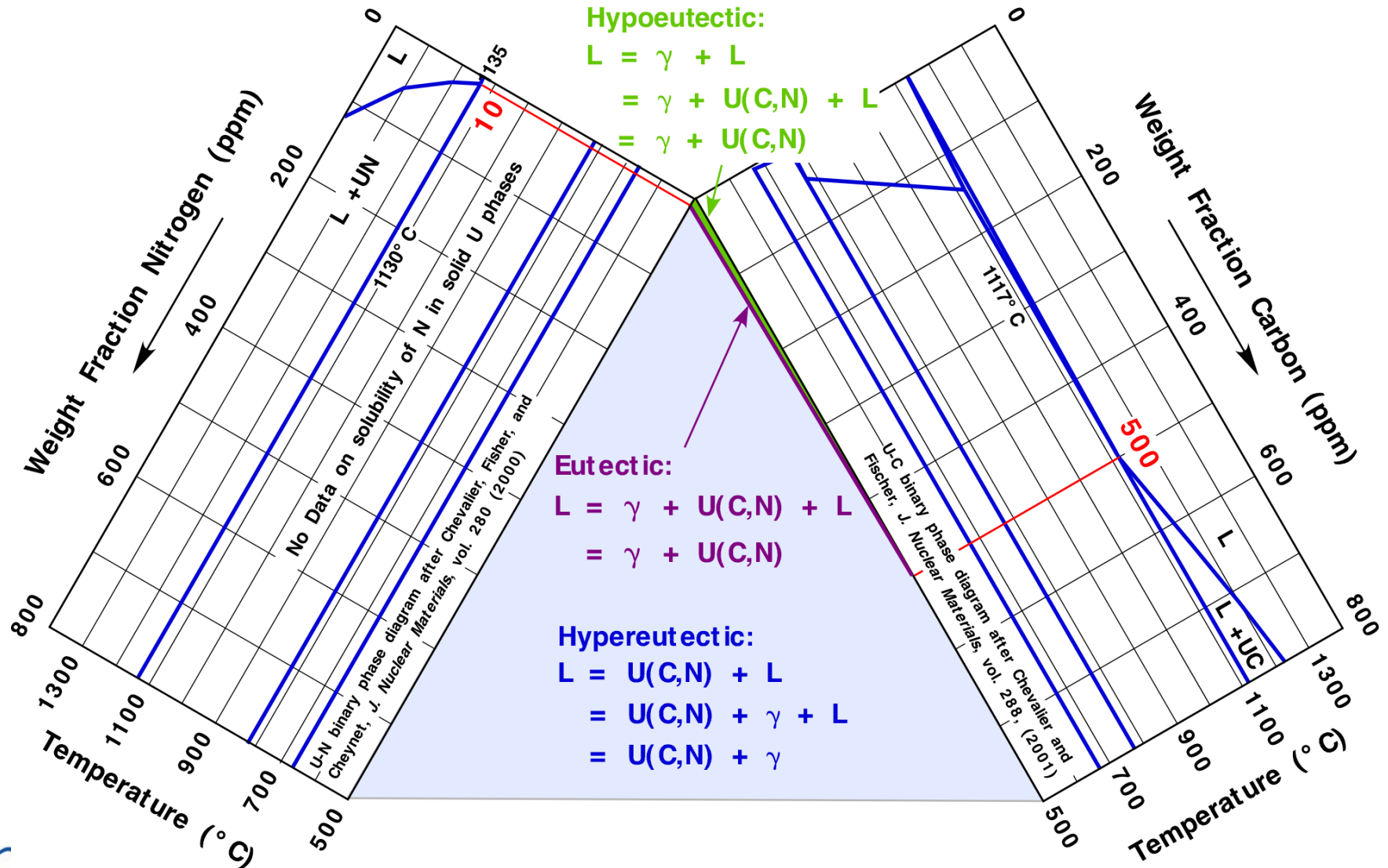


Optical micrographs of as electro-polished samples
(27% phosphoric acid - 27% ethylene glycol - ethanol)



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Conceptual U-C-N Phase Diagram



Large Single-Zone VIM Furnaces: E and F-Furnace

E & F Furnaces:

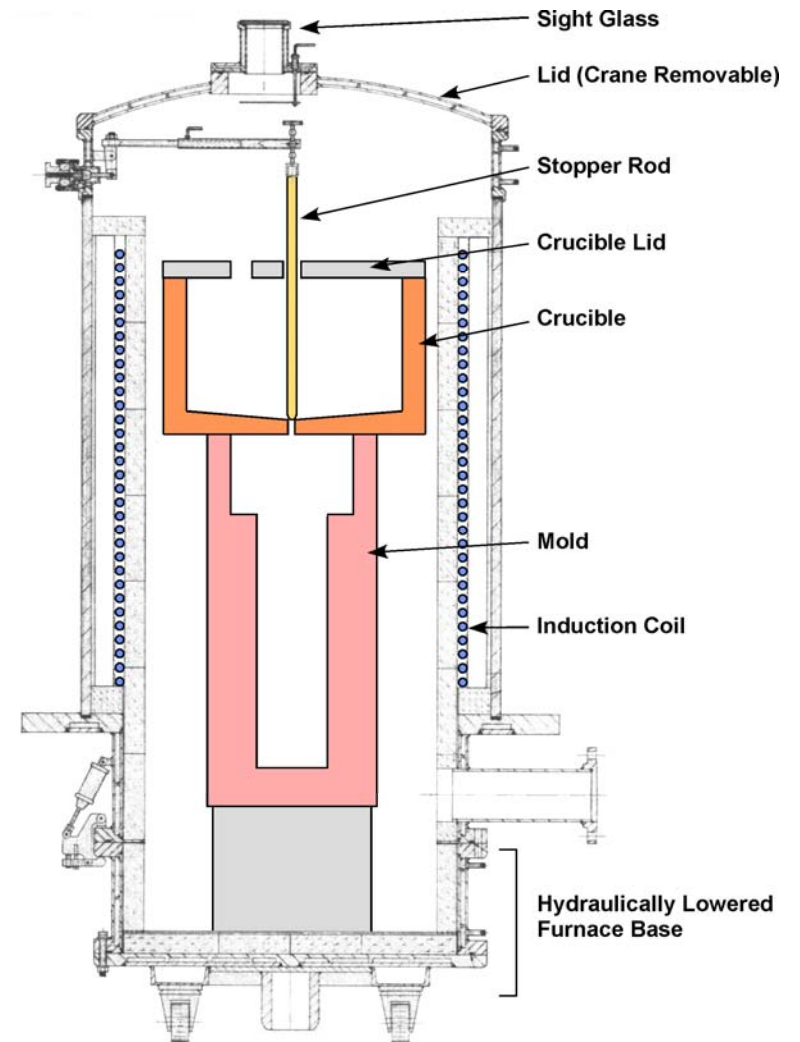
- Shared 250 kW / 1 kHz power supply
- Similar to H-wing furnaces at Y-12

E Furnace:

- 1000 kg of DU
- Coil 46" x 35" ϕ

F Furnace:

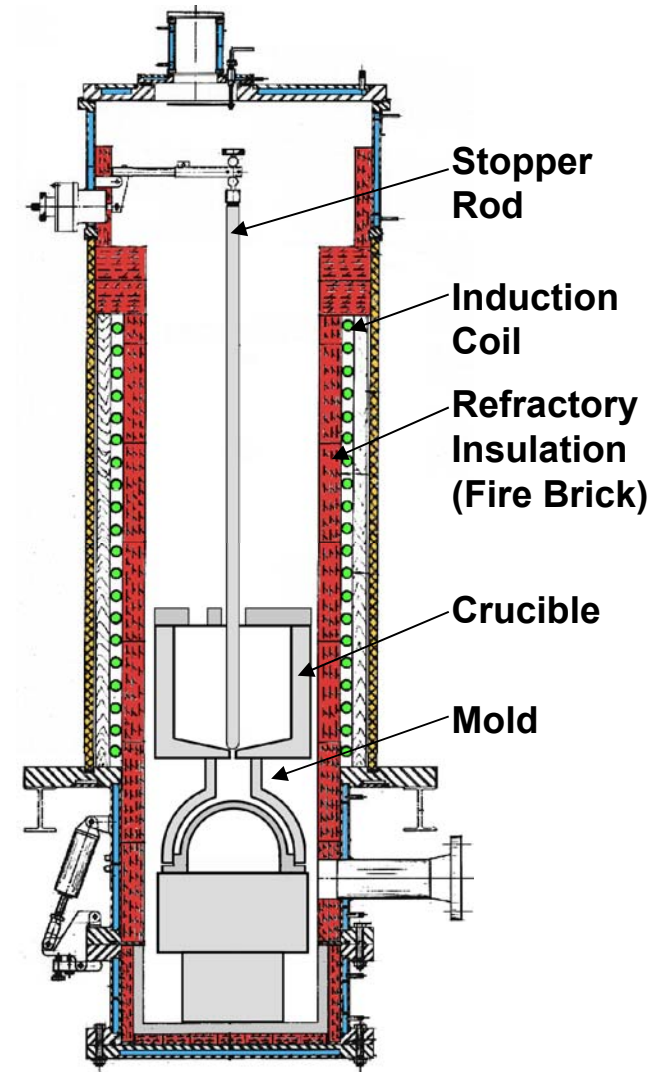
- 2500 kg of DU
- Coil 36" x 48" ϕ



Single-Zone Vacuum Induction Furnace: C-Furnace

Single induction coil:

- 200 kg of DU capacity
- Coil 36" x 18" ϕ
- 100 kW at 3 kHz
- Inductotherm Power-Trak 100-30 power supply

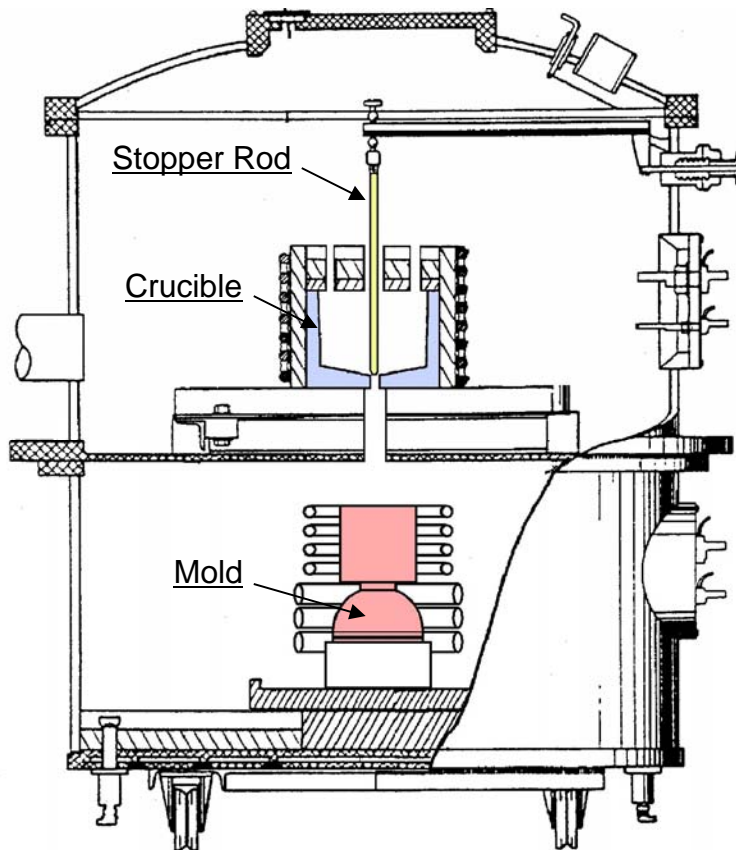


C, C1, and D are identical designs using shared power supply and pumps

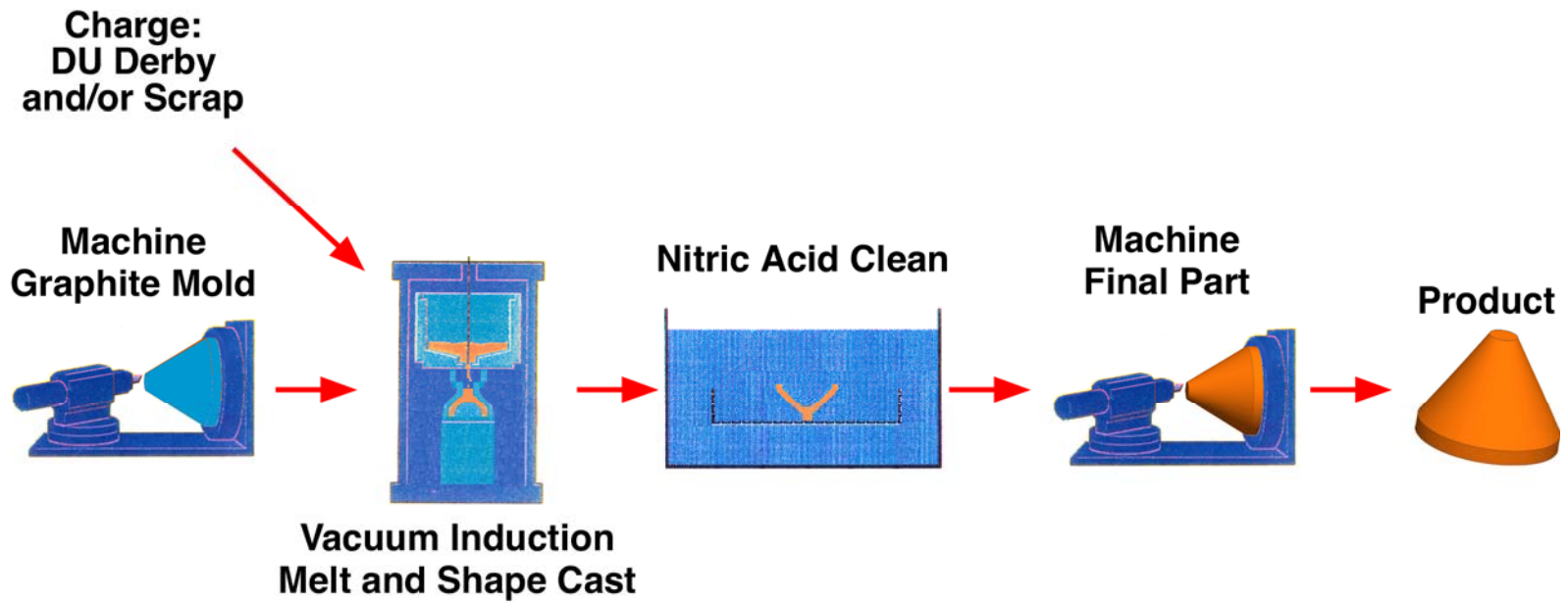
LANL Three Zone VIM: K Furnace

Three separately controlled induction coils:

- Melting crucible coil (35 kW at 9.6 kHz)
- Two crucible coil sizes (14" and 18" diameter)
- Two mold heating coils (50 kW at 3 kHz)
- Three mold heating coil sizes (11", 14", and 22" diameter)

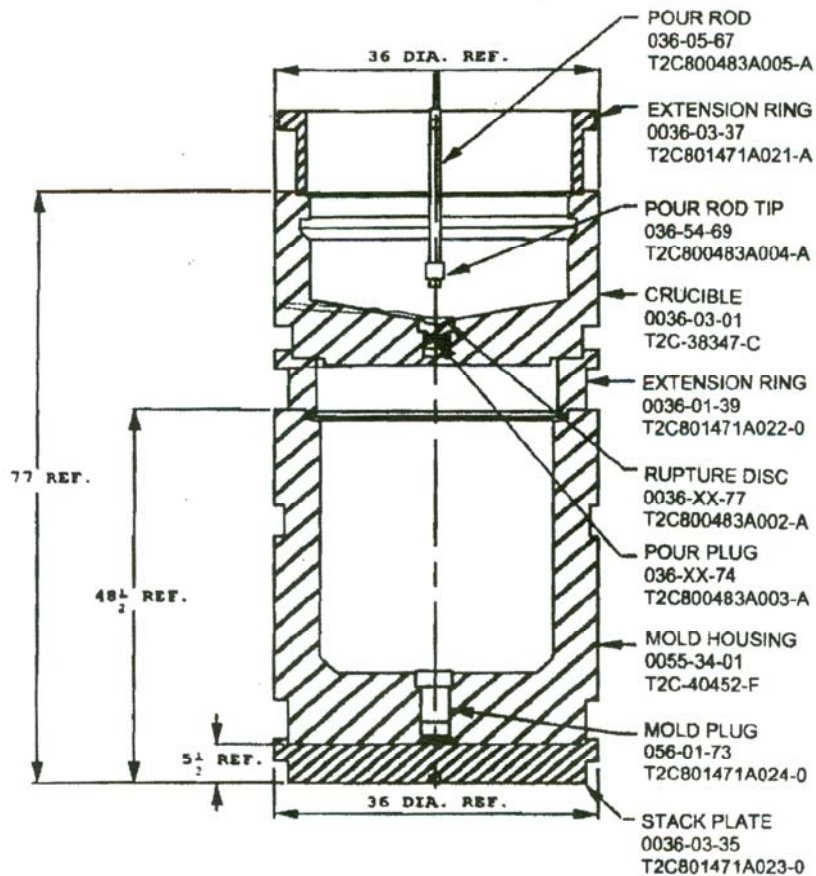


DU Shape Casting

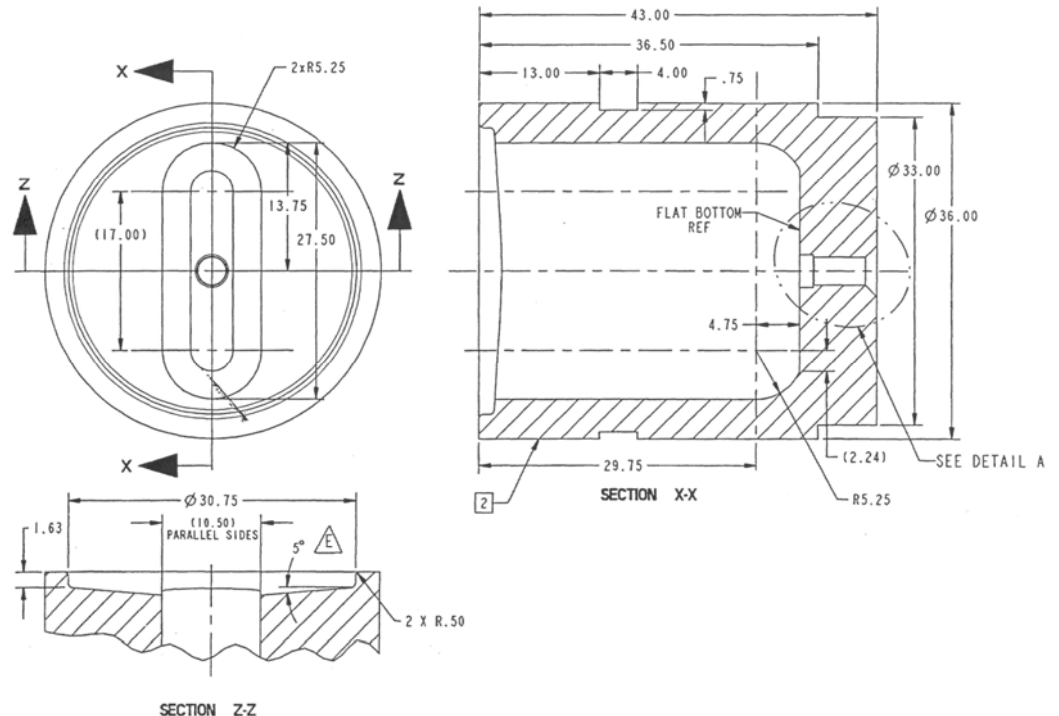


Casting of Large 2200 kg Rolling Billets

Mold Stack

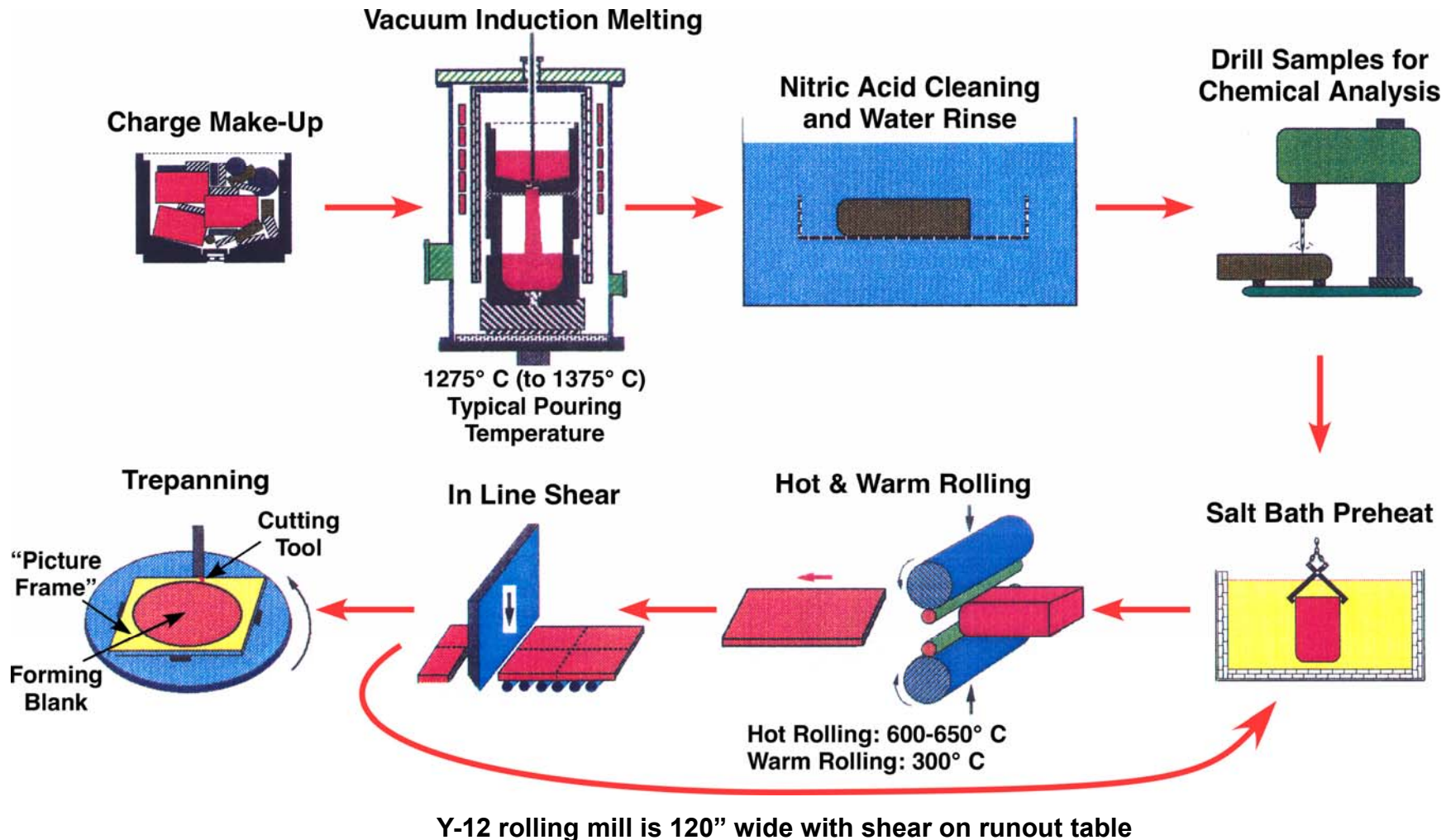


Rolling Billet Mold

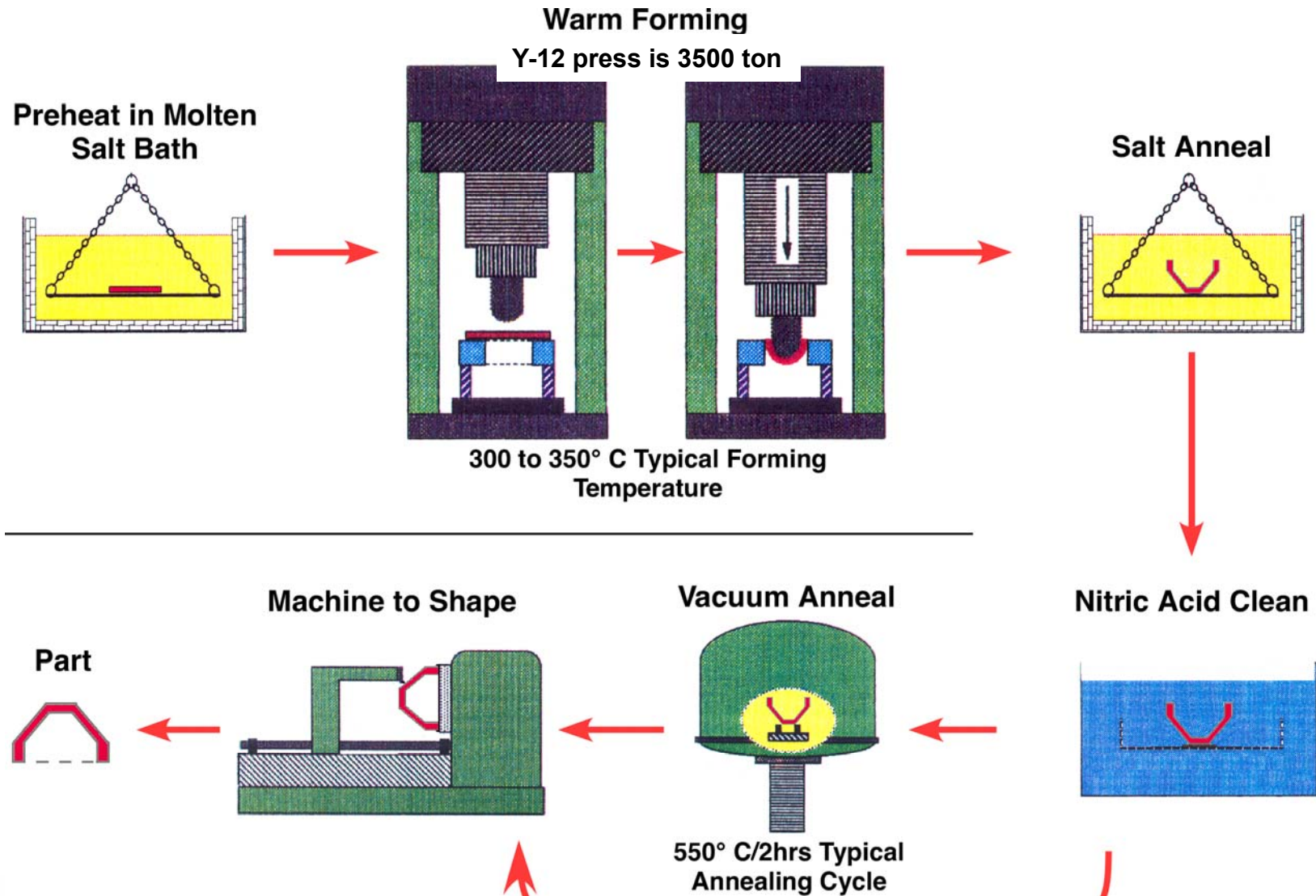


- Large hot crucible sitting on top of mold helps with directional solidification and eliminates need for a hot top
- Historic Y-12 procedure calls for heating to 1275°C, hold for 20 minutes, and pour

DU Casting and Wrought Processing (1)



DU Casting and Wrought Processing (2)



DU Hydro Plate (1)

- **Hydro Plate is a 0.3” thick fine-grained low-carbon controlled-texture wrought DU used for hydrodynamic testing**
- **Historically produced under specification SP-Y-007**
 - **Chemistry tightly controlled**
 - **Rolling sequence and temperatures leaves room for variation**
- **Current specification is RM6K0374**
 - **Chemistry is tightly controlled**
 - **Rolling sequence is specified to try to control crystallographic texture**
 - **Specific to 0.3” thick final thickness plate**
- **Plate procured in the warm worked state (strain greater than critical recrystallization strain)**
- **Components formed then recrystallized (550°C) before use to produce a fine uniform grain size**

DU Hydro Plate (2)

Rolling sequence is specified to try to control crystallographic texture

- **Recommended hot rolling schedule from 10" to 0.9" provided**
- **Warm rolling schedule from 0.9" to 0.3" must be followed**

	Pass	Nominal Thickness		Actual Thickness		Rolling Direction
		(mm)	(in.)	(mm)	(in.)	
Hot Rolling 635°C ± 30°C	initial	254.00	10.000	254.00	10.000	
	1	226.62	8.922			0°
	2	202.18	7.960	202.18	7.960	0°
	3	173.55	6.833			90°
	4	148.97	5.865	148.97	5.865	90°
	5	131.43	5.174			0°
	6	115.95	4.565	115.95	4.565	0°
	7	90.00	3.543			90°
	8	69.85	2.750	69.85	2.750	90°
	9	53.28	2.098			0°
	10	40.64	1.600	40.64	1.600	0°
	11	35.20	1.386			90°
	12	30.48	1.200	30.48	1.200	90°
	13	26.40	1.039			0°
	14	22.87	0.900	22.87	0.900	0°
Anneal 635°C ± 10°C						
Initial Warm Working 330°C ± 30°C	initial	22.87	0.900	22.87	0.900	
	1	20.67	0.814			0
	2	18.67	0.735			315°
	3	16.87	0.664			270°
	4	15.25	0.600			225°
	5	13.78	0.542			180°
	6	12.45	0.490			135°
	7	11.25	0.443			90°
	8	10.17	0.400	10.17	0.400	short side
Anneal 635°C ± 10°C						
Final Warm Working 330°C ± 30°C	initial	10.17	0.400	10.17	0.400	
	1	9.46	0.372			90°
	2	8.80	0.347			45°
	3	8.19	0.323			0°
	4	7.62	0.300	7.62	0.300	short side

Chemistry Limits of Common Unalloyed DU Specifications

DU Chemical Specifications - Maximum ppm				
Element	High-Purity, High-Ductility, Fine Uniform Structure Uranium Parts	High Purity Wrought DU for Hydro Test Assemblies	Wrought Depleted Uranium Plate Specification	Depleted Unalloyed Uranium Specification
	SP-Y-007	RM6K0374	RM6K0065	RM6K0049
Al	15	15	50	
Be				10
B				10
Ca	50	50	75	
C	100	100	200	475
Cu		25	50	
Fe	75	75	200	300
Li				10
Mg	5	5	75	
Mn	35	35	50	
Mo		25	150	105
Ni	50	50	100	115
Nb		25	25	
S				10
Si	75	75	150	475
Ti		25	25	
V		5	25	
Zr		25	100	

Ingots Casting from Derby

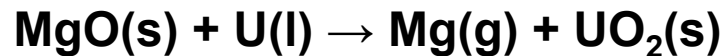
*From C.D.Harrington and A.E. Ruehle, Uranium Production Technology,
D. Van Norstrand Co., NJ, p. 259 (1959)*

After the furnace is loaded and sealed as described above, it is evacuated by vacuum pumping. When the pressure has dropped to 200 microns, induction heating is started. Pumping and heating are continued until the melt temperature reaches 1454°C (2650°F). (The melting point of uranium of 99.99 percent purity is given [20] as $1133 \pm 1^\circ\text{C}$ (2071.4°F).) At this temperature, the melt is allowed to outgas for 45 minutes. Magnesium, some slag, hydrogen, radioactive decay products such as UX₁, and other volatile impurities distill from the melt. The power is then turned off, the furnace is isolated from the pumping system, and the pour is made by manually actuating the pouring mechanism which breaks the graphite plug. About 2 minutes after the pour is

Removal of Deleterious Inclusions

During casting hypo-eutectic inclusions may be removed by

- Chemical Reactions



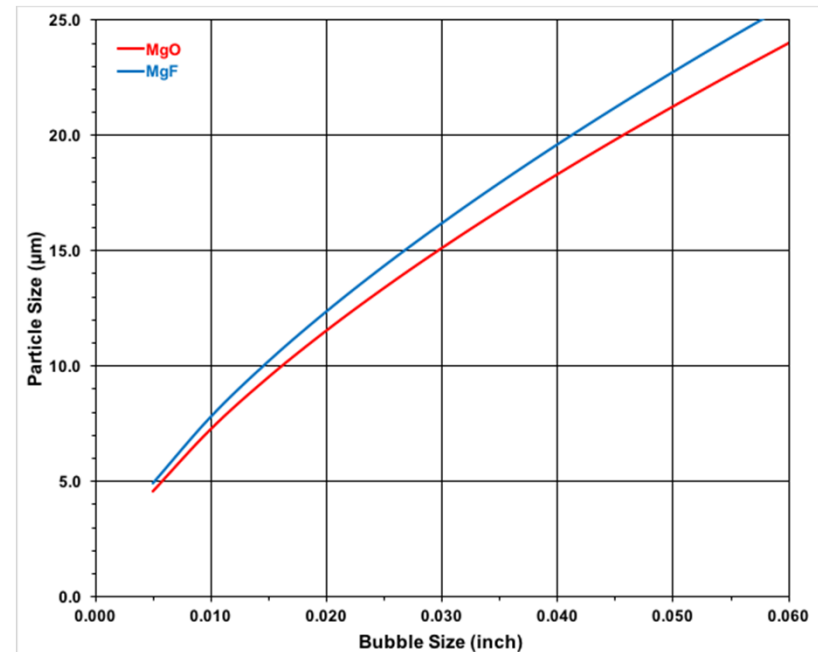
- Floatation

Stokes law gives flotation velocity of a spherical inclusion

$$V = \frac{2}{9} \frac{gR^2}{\mu} (\rho_{\text{particle}} - \rho_{\text{liquid}})$$

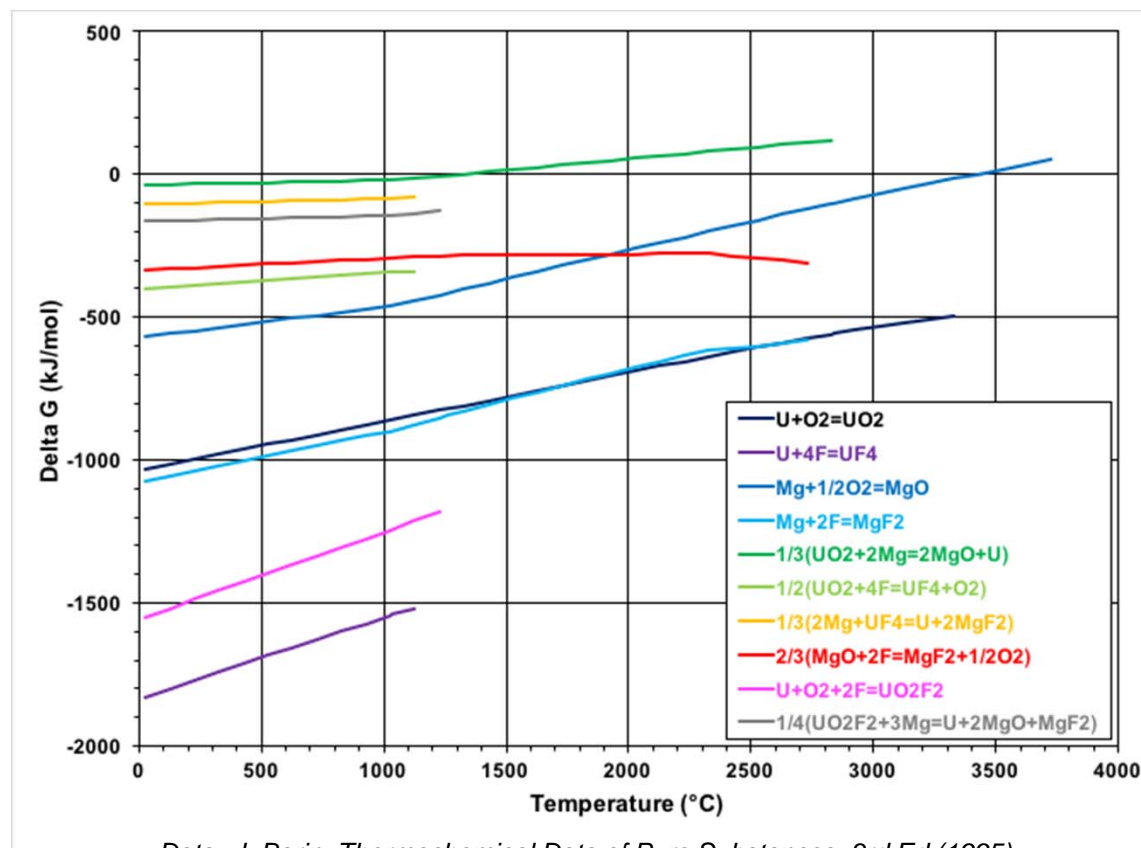
Additionally

- High vapor pressure elements can volatilize (Mg, Na, K, F)



Thermodynamics of U-Mg-F-O System

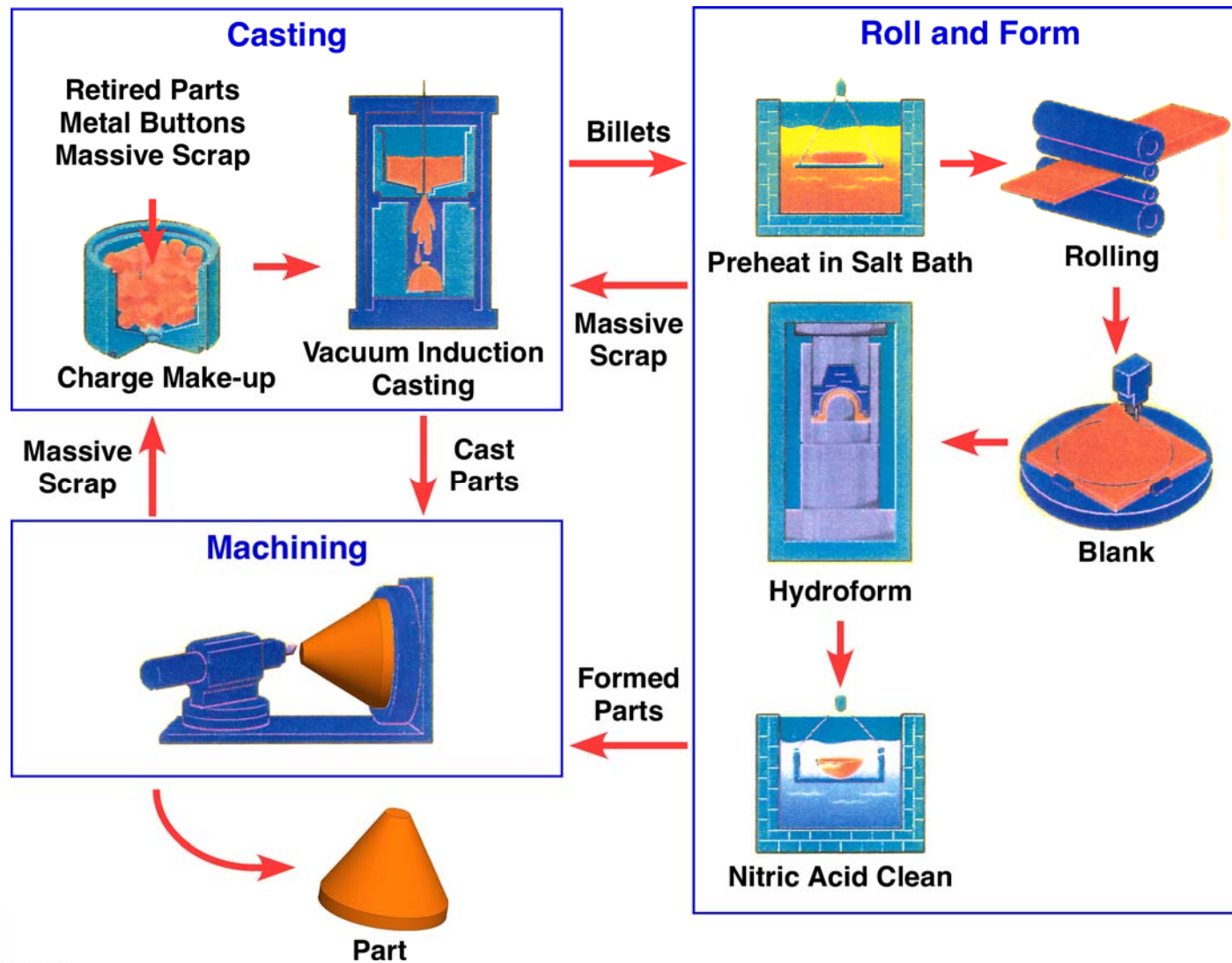
- Above 1303°C MgO is reduced
 $\text{MgO(s)} + \text{U(l)} \rightarrow \text{Mg(g)} + \text{UO}_2\text{(s)}$
- Above 3420° C MgO decomposes
 $\text{MgO(s)} \rightarrow \text{Mg(g)} + 1/2\text{O}_2\text{(g)}$



Data - I. Barin, Thermochemical Data of Pure Substances, 3rd Ed (1995)

Processing of Enriched Uranium Alloys

Enriched Unalloyed Uranium Casting and Forming

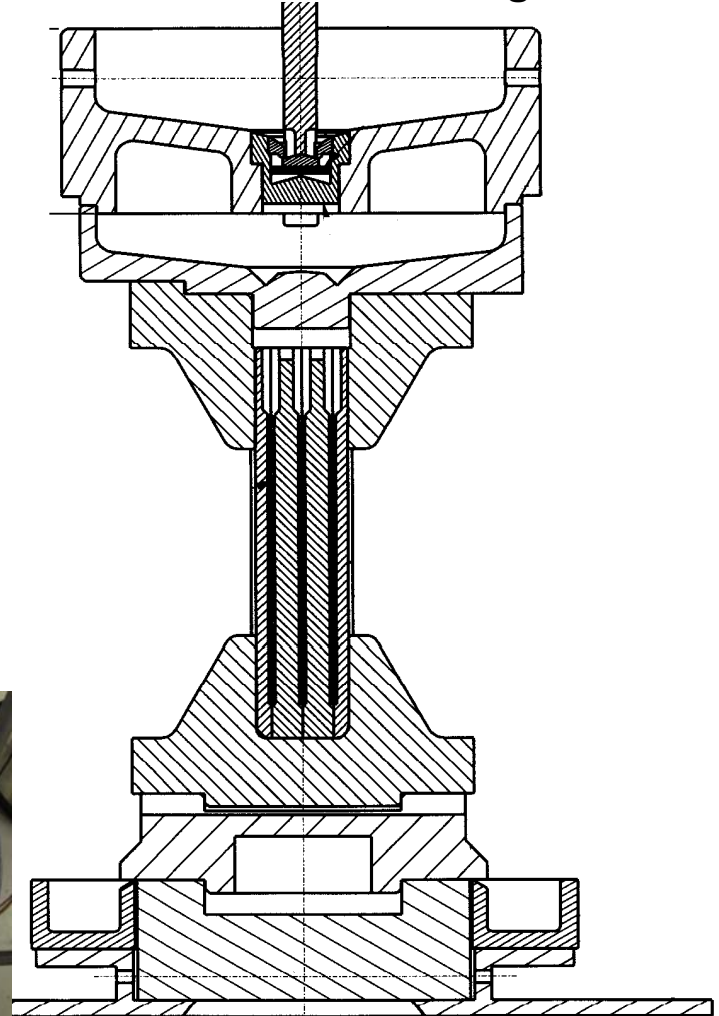


Enriched Uranium Casting

- Major difference from DU processing is charge limitations due to criticality concerns
 - Y-12 is currently limited to 28 kg
 - UPF castings will be limited to 22 kg
- For wrought EU this means very small rolling billets and less than necessary wrought reductions
- Incorporation of mold features to keep metal in “known” geometry
- Incorporation of features mitigate for human error
 - Crucible has over-flow holes to prevent over charging

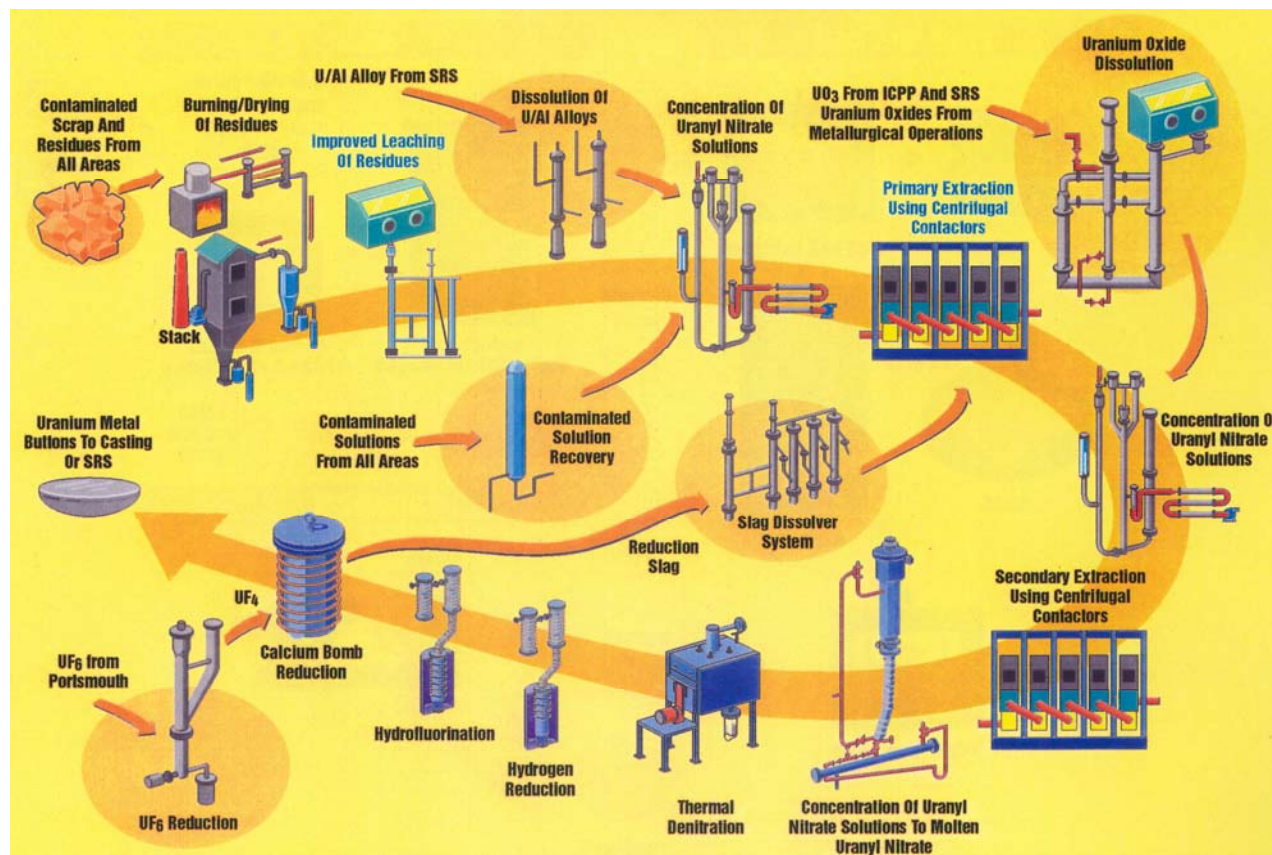


Y-12 T802077-0016 Mold for LEU-10Mo Plate Castings



HEU Chemical Reprocessing

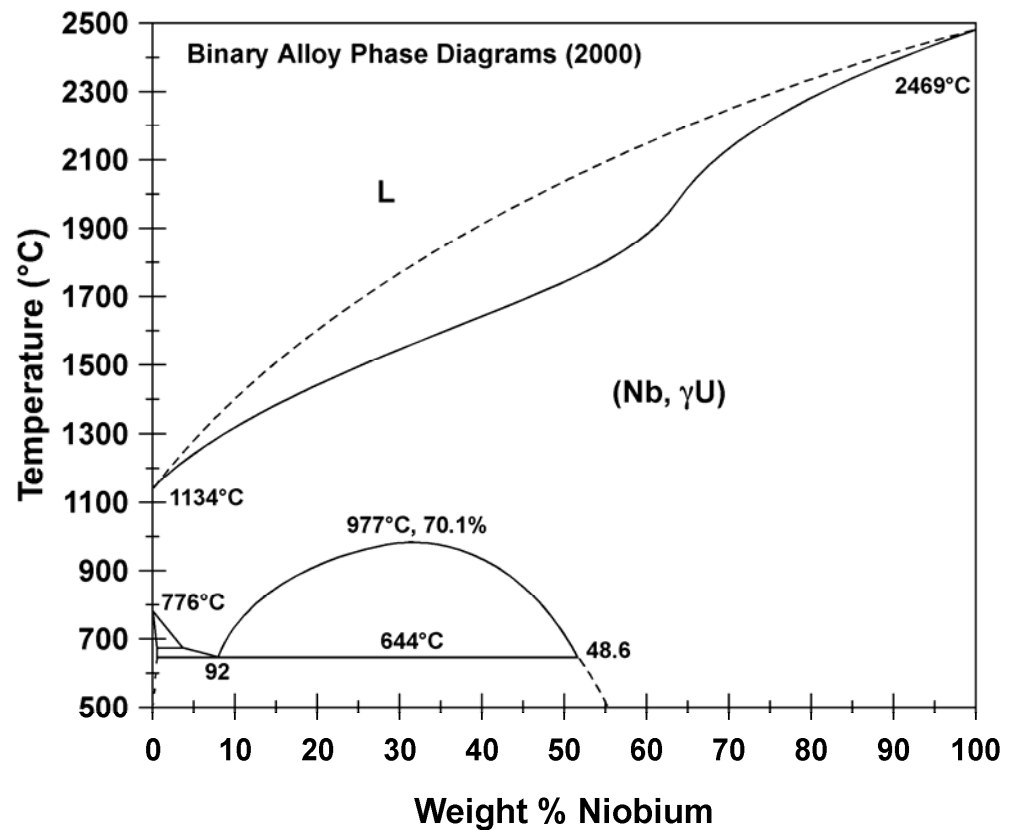
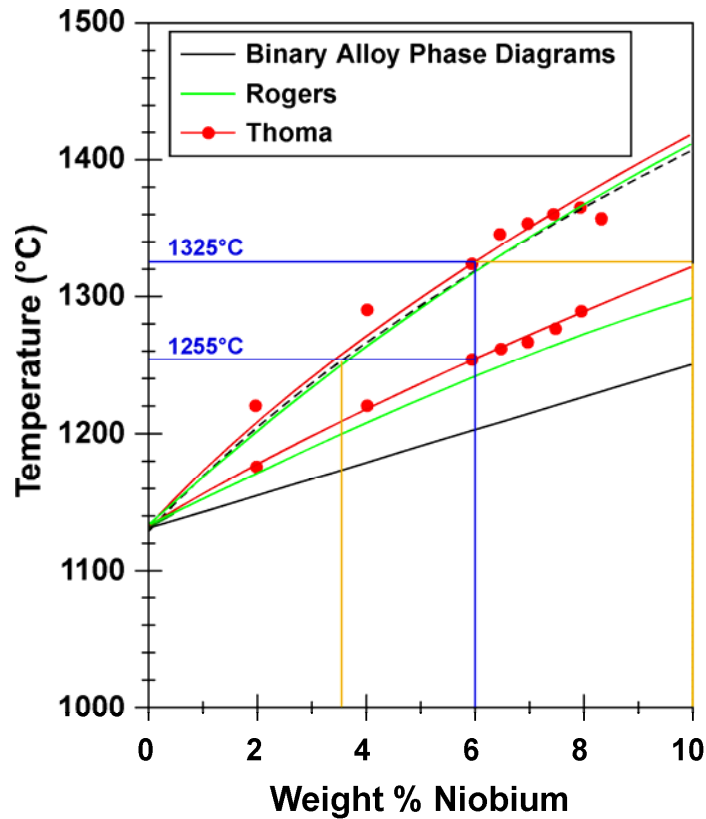
- The high cost of enriched uranium means that material recycling is an important component of the process
- Historically used complex chemical reprocessing
- Future UPF facility will use electro-refining



Processing of U-Nb Alloys

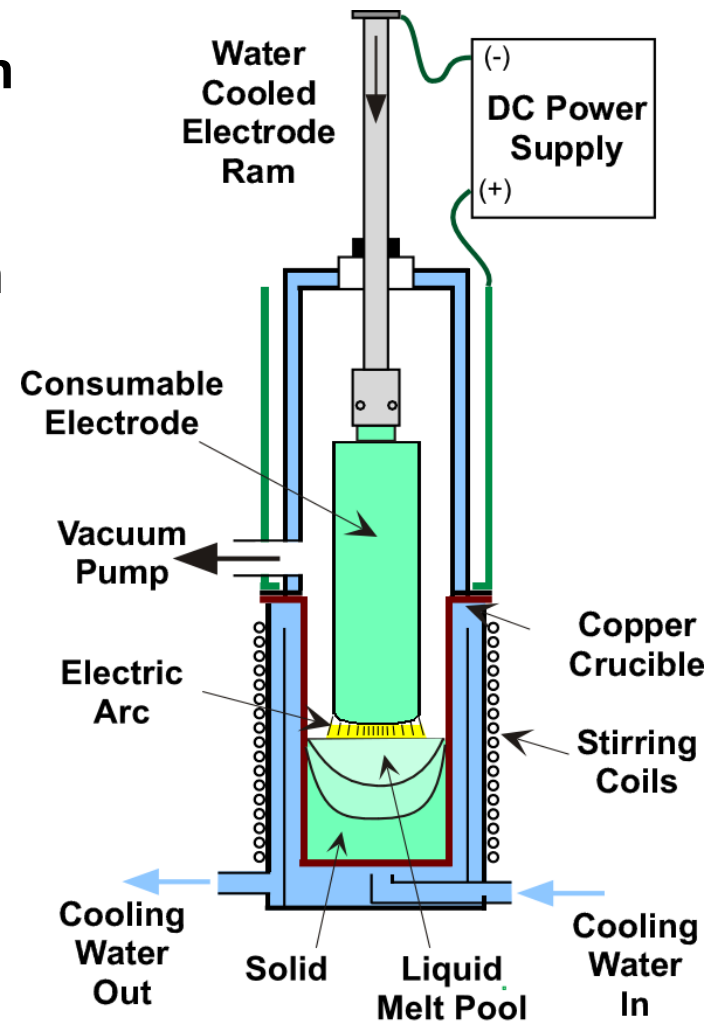
- **Wrought U-6Nb**
 - Vacuum Arc-Remelting (VAR)
 - U-6Nb Ingot Production (and Variations)
 - Wrought Processing
 - U-6Nb Macrostructure

U-Nb Phase Diagram



The Vacuum Arc Remelting (VAR) Process

- Vacuum Arc Remelting (VAR) is a melting process used in the production cylindrical ingots of various premium metals and alloys
- A continuous DC arc is struck between an electrode, of the metal to be melted, and a cooled copper crucible
- Heat from arc melts the electrode and provides heating into the top of the ingot/melt pool
- Molten metal drips from electrode to the bottom of the crucible and solidifies into an ingot
- As electrode melts back and ingot grows the electrode is fed in



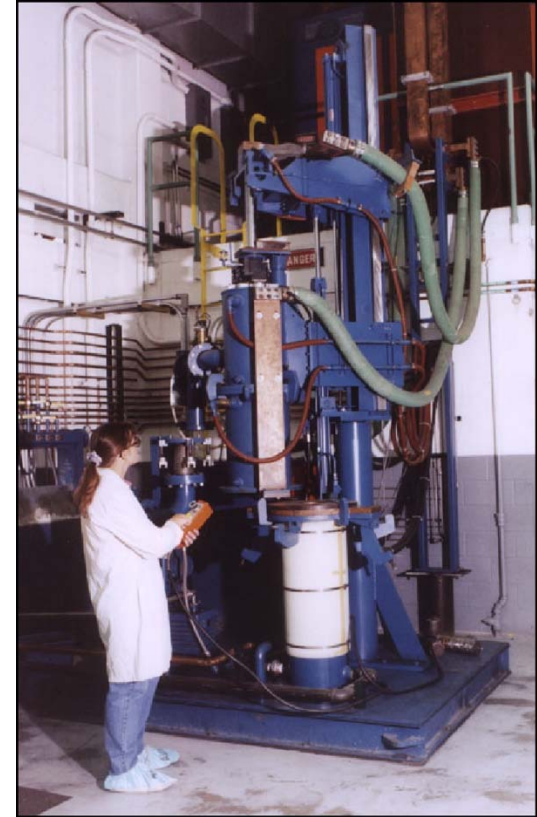
Why VAR is Used Industrially for Ingot Production

Primary benefits of remelting a consumable electrode under vacuum are:

- 1) Localized heat input allows melting of high melting point metals and alloys with high melting point constituents**
- 2) Vacuum processing/refining**
 - Removal of dissolved gases such as hydrogen, nitrogen, and CO
 - Reduction of undesired trace elements with high vapor pressure
- 3) Cold Hearth Melting Process**
 - No contamination from ceramic crucible or mold
 - High heat extraction rate
 - Finer dendrite arm spacing
 - Minimize micro-segregation
- 4) Achievement of directional solidification of the ingot from bottom to top, thus avoiding macro-segregation and reducing micro-segregation**

VAR Equipment – LANL Research VAR

- Configured for melting reactive metals with remote operators console
- Designed and build by Retech, installed in 1983
- New custom controller installed 2003; PLC updated 2015
- 10 kA power supply
- 2.5", 3.5", 5", 6", 6.25" and 8.5" diameter x 24" crucibles; up to 400 kg
- Magnetic stirring up to 90 G
- Highly instrumented including load cell and video recording; instrumented crucibles (temperature and current partitioning)
- Both voltage controller and Sandia National Laboratory/Specialty Metals Processing Consortium developed melt-rate/solidification-rate controller



VAR Equipment – Dual Station VAR Furnaces



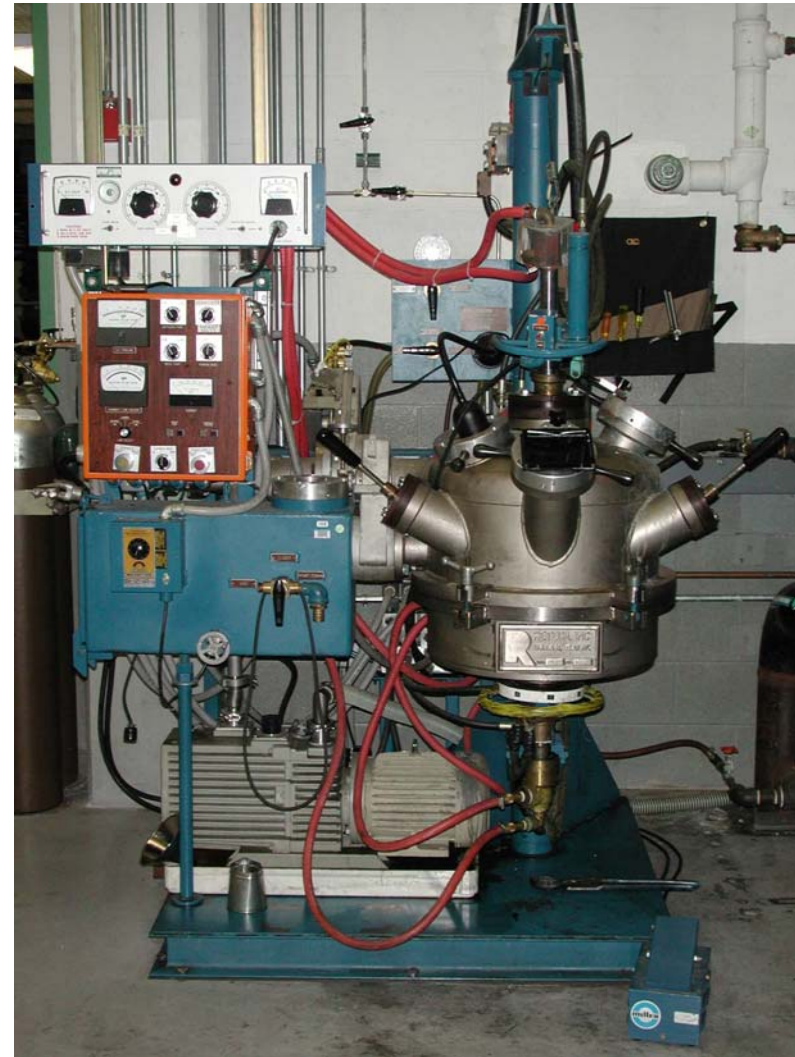
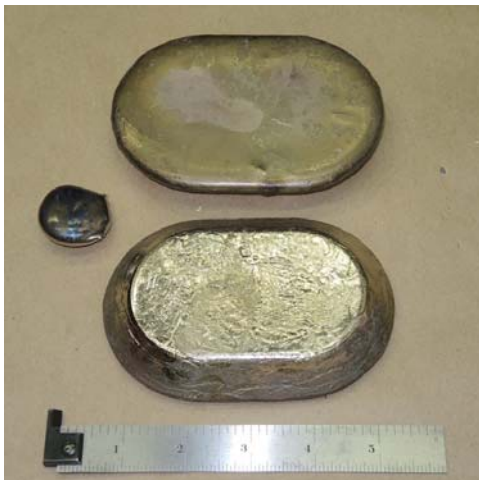
Large Retech Furnace



Consarc 28" Furnace

Non-Consumable Arc Melter

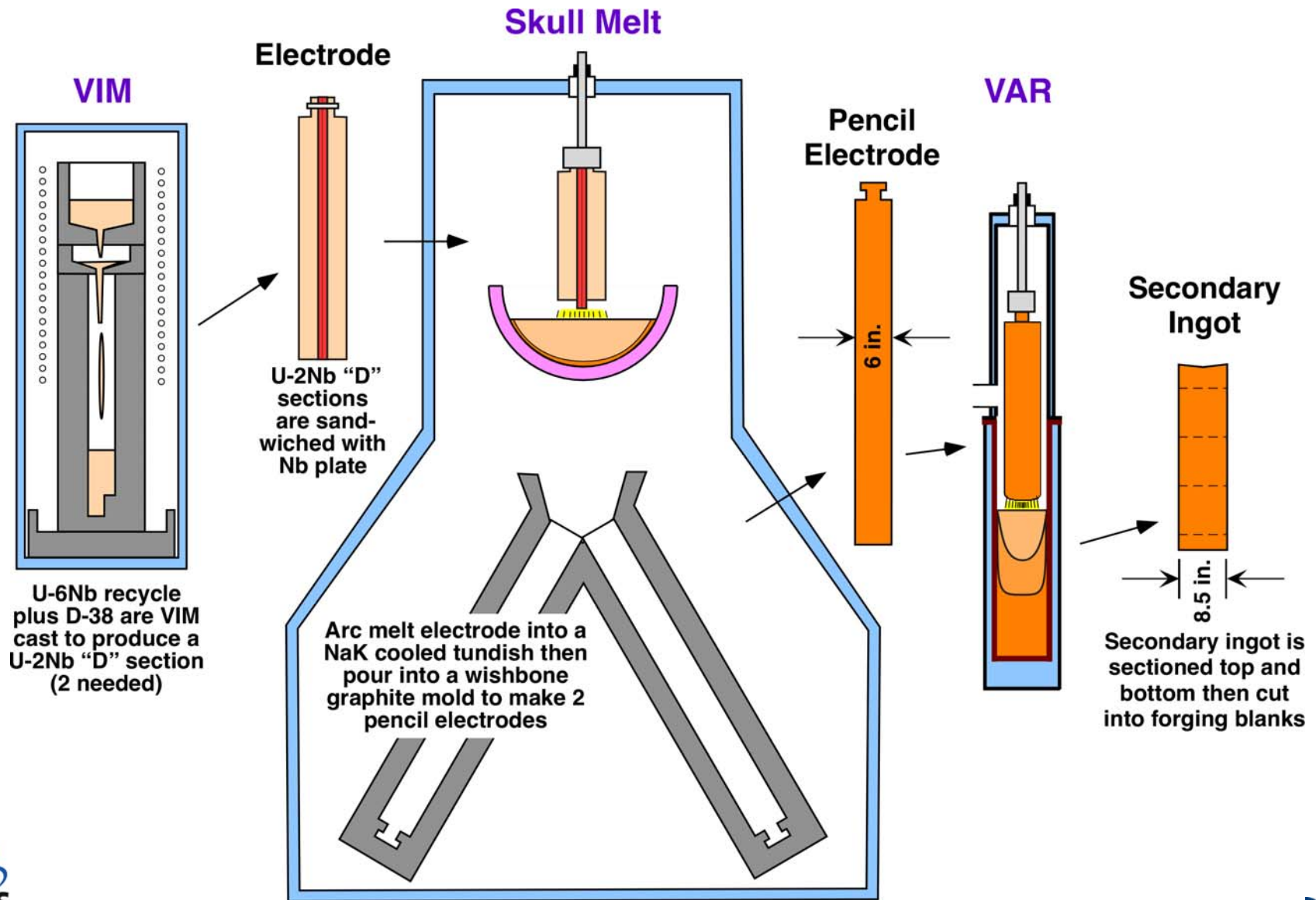
- Used for initial alloying of materials with highly dissimilar melting points and producing small samples of high melting point metals/alloys
- Uses a arc struck between a non-consumable electrode and button to melt the metal in a copper hearth
- A variety of hearth pocket sizes
- 1.6 kA power supply



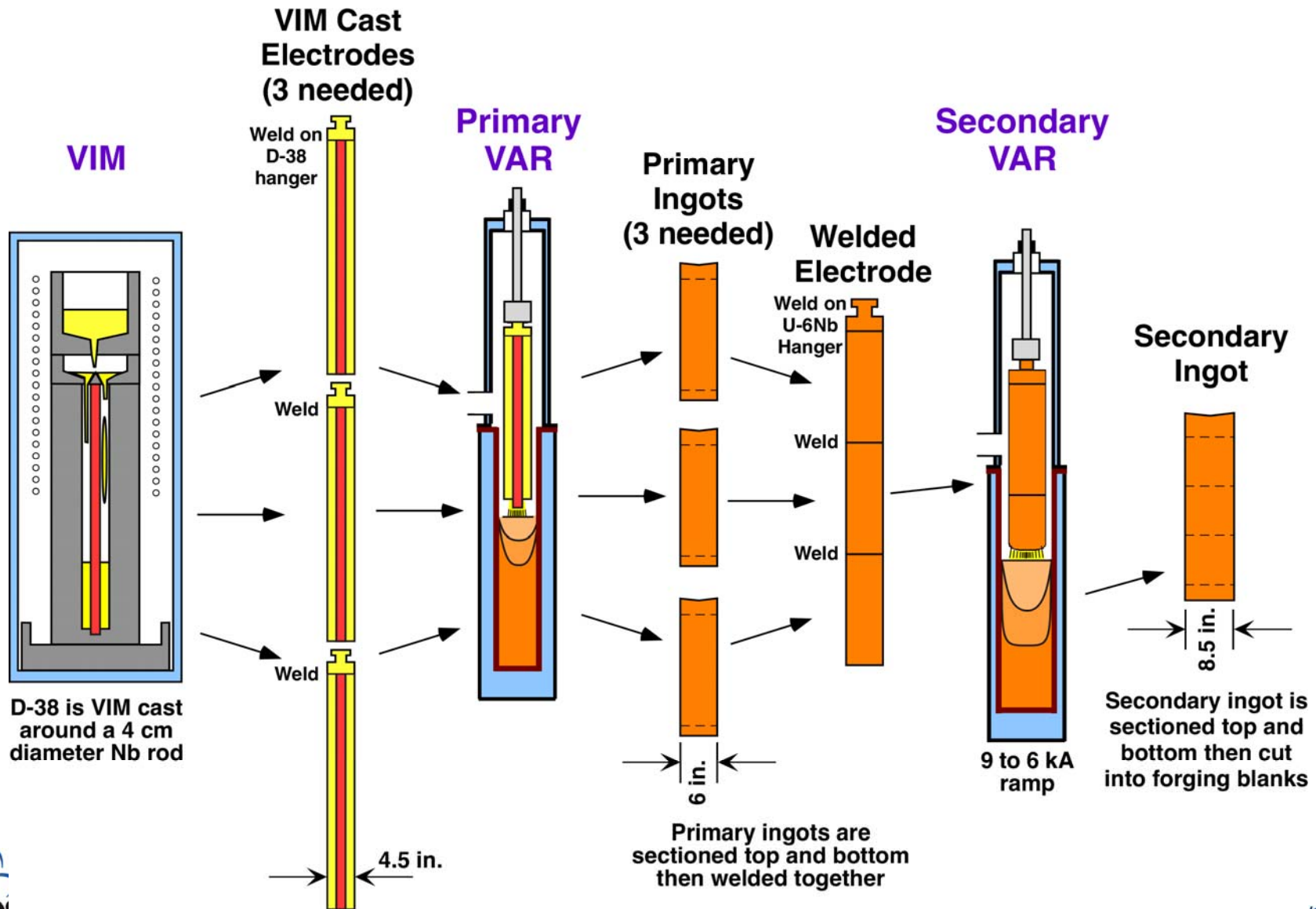
Safety – How Worried Should We be about VAR Processing of Uranium?

- **Ti and Ti Alloys**
 - Large melt pool (2,000 to 10,000 kg)
 - High current (40 to 80 kA)
 - Will crack steam into H_2 (-479 kJ/mol: $2H_2O + Ti = TiO_2 + 2H_2$)
 - Sketchy primary electrode (welded together from sponge and scrap)
- **U and U Alloys**
 - Small melt pool (100 kg) for VAR
 - Large melt pool (1500 kg) for Skull melting
 - Low current (4 to 9 kA)
 - Will crack steam into H_2 (-598 kJ/mol: $2H_2O + U = UO_2 + 2H_2$)
 - Sound electrode (VIM cast primary or welded VAR secondary)
- **Ni Superalloys**
 - Small melt pool (400 kg)
 - Low current (10 kA)
 - Will not crack steam into H_2 (+4 kJ/mol: $H_2O + Ni = NiO + H_2$)
 - Sound electrode (VIM or ESR)

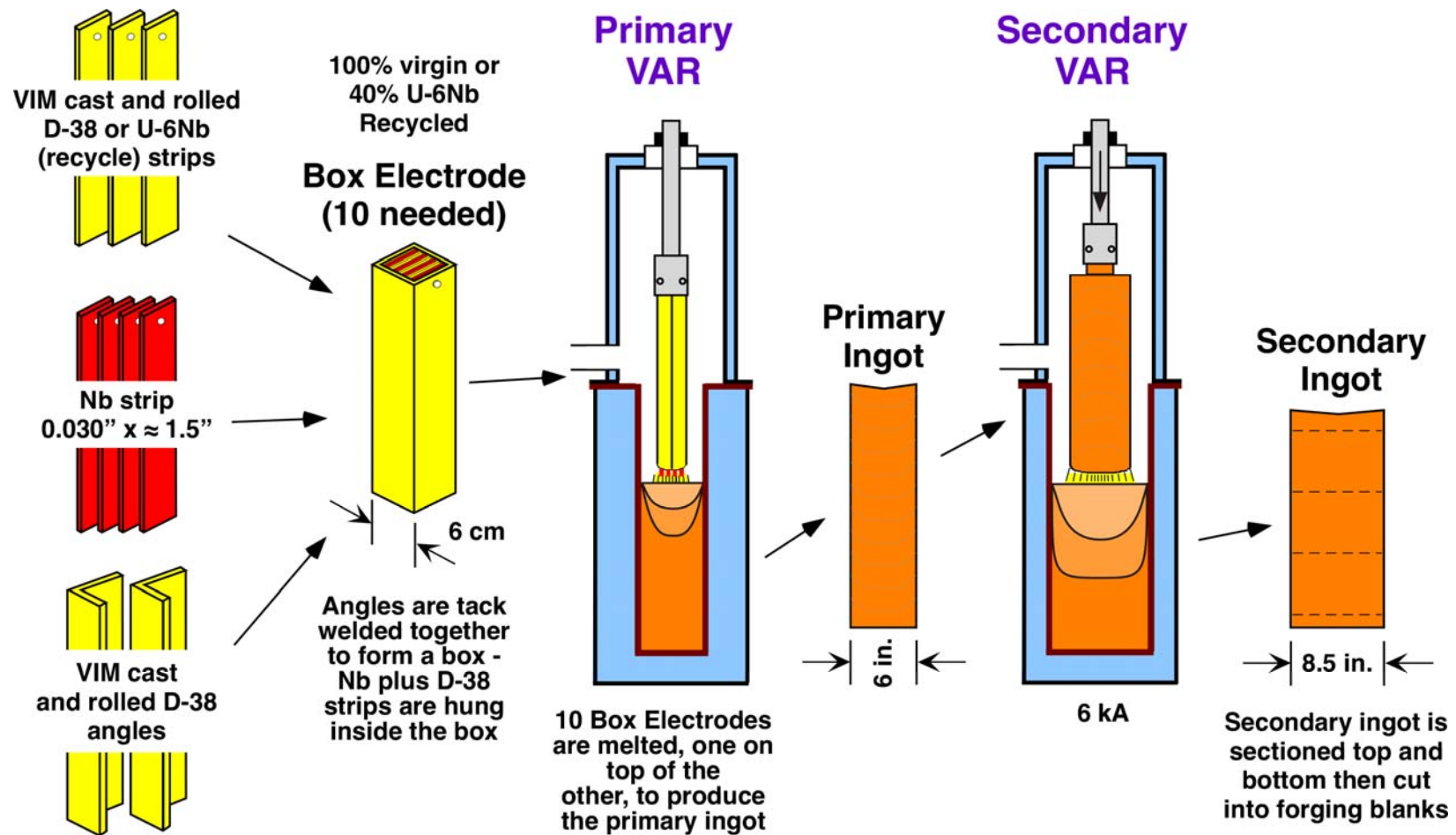
Historic Y-12 VIM-Skull-VAR Process for U-6Nb



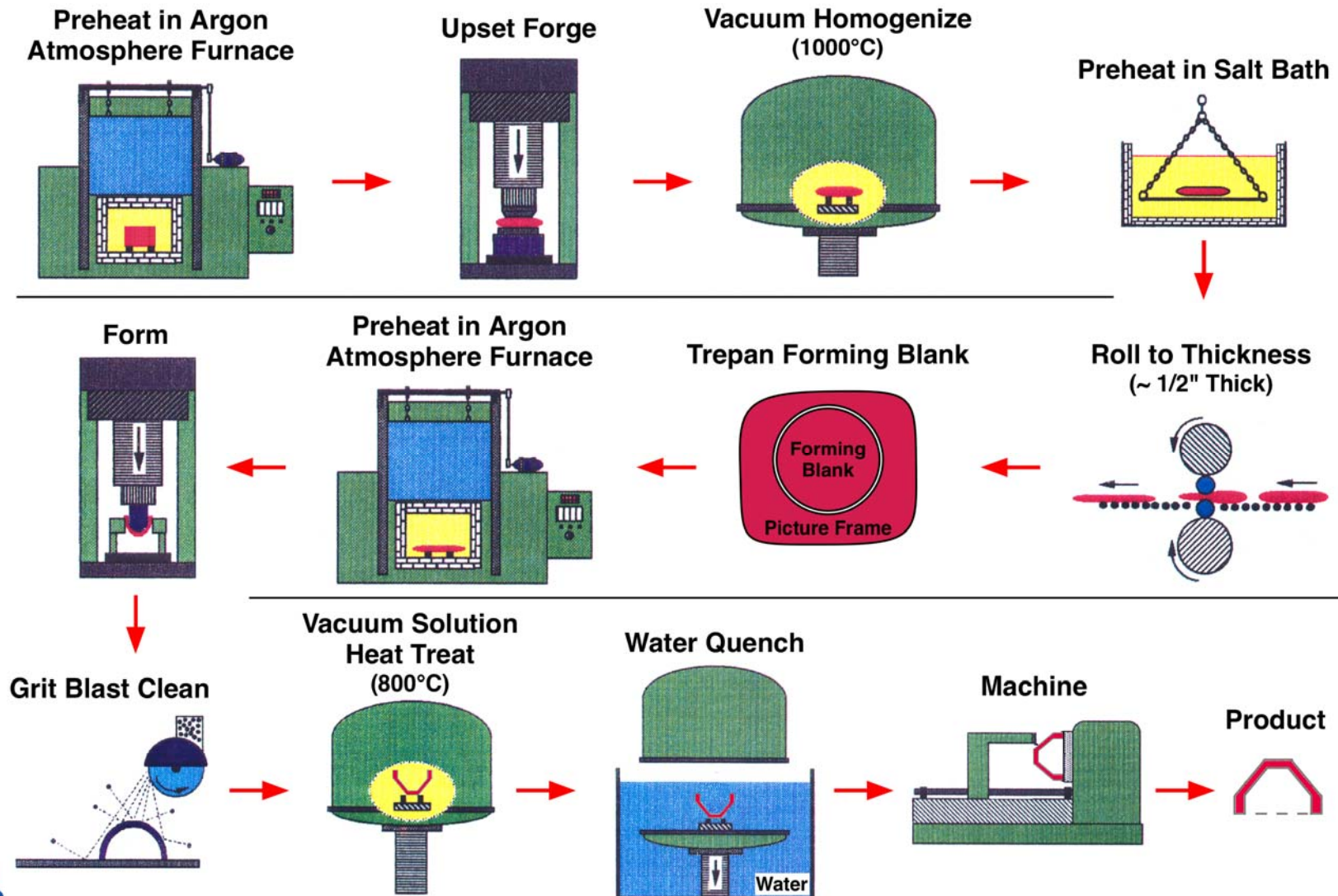
Y-12 VIM-VAR-VAR Process for U-6Nb Ingot



Rocky Flats Box Electrode VIM-VAR-VAR Process for U-6Nb Ingot



Wrought Processing of U-6Nb Billet



U-6Nb Specification OO-M-200/OO-M-207

Key requirements of the wrought U-6Nb specification:

Chemical Composition: The chemical composition of the material shall be as follows:

	<u>Minimum by wt</u>	<u>Maximum by wt</u>
Uranium (depleted)	93.0%	----
Niobium	5.2%	6.5%
Carbon	----	200 ppm
Copper	----	100 ppm
Iron	----	150 ppm
Nickel	----	75 ppm
Aluminum	----	75 ppm
Silicon	----	150 ppm
Molybdenum	----	75 ppm
Titanium	----	75 ppm
Zirconium	----	500 ppm
Tantalum	----	200 ppm

Mechanical Properties: The mechanical properties of the parts shall be as follows:

- 4.5.1 **Unaged:** The part shall be solution annealed and quenched from the gamma field.
The mechanical properties for material in this condition shall be:

	<u>Minimum</u>	<u>Maximum</u>
Ultimate Tensile Strength	700 MPa (101.5 ksi)	---
Tensile Yield Strength* at 0.2% offset	100 MPa (14.5 ksi)	200 MPa (29 ksi)
Elongation	25 %	---

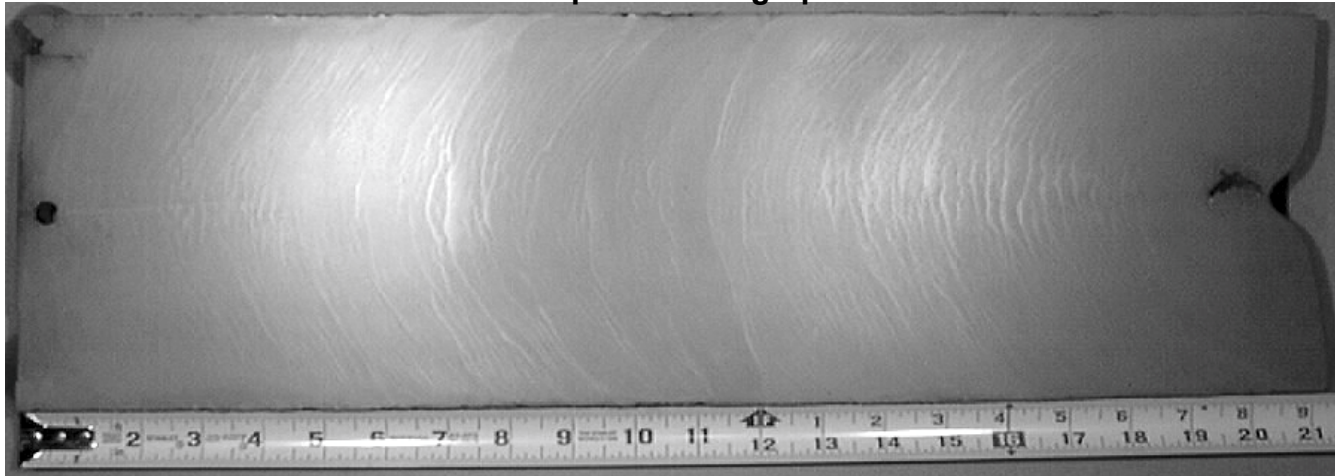
* Assuming an elastic modulus of 69 GPa (10,005 ksi)

U-6Nb Going Forward

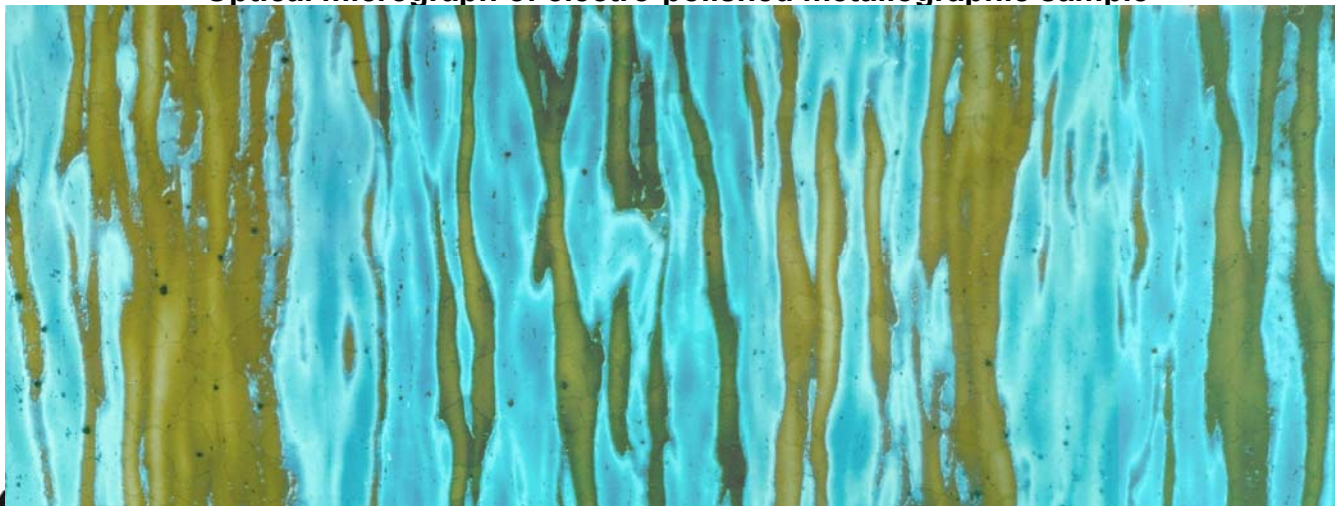
- **Historic VIM-Skull-VAR Process**
 - 4% efficient
 - 30% recycle of material
 - DU derby was free
- **Current VIM-VAR-VAR Process**
 - 4% efficient
 - No material recycle
 - DU derby will be ~\$100/kg
- **Material cost and the large number of processing steps makes wrought U-6Nb very expensive and has prompted considerations of other approaches**

Niobium Banding in VAR Melted U-6%Nb

U-6Nb VAR ingot cross-section
Optical micrograph

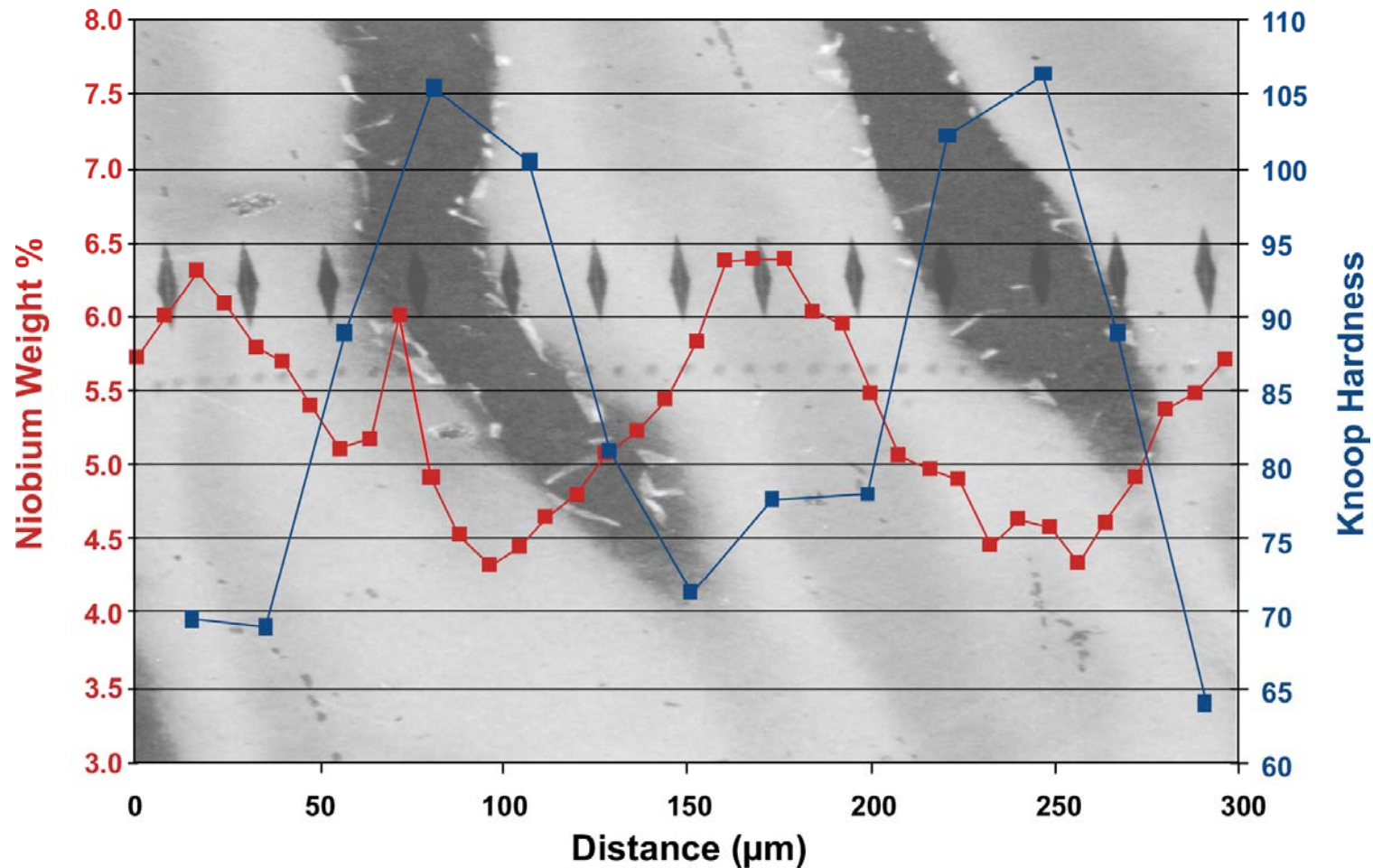


U-6Nb VIM-Skull-VAR ingot rolled to plate and heat treated
Optical micrograph of electro-polished metallographic sample



Segregation and Banding in U-6%Nb Ingot

U-6Nb VIM-Skull-VAR ingot rolled to plate and heat treated



Macro Banding in U-6Nb by Various Processes

Quantitative Trend -

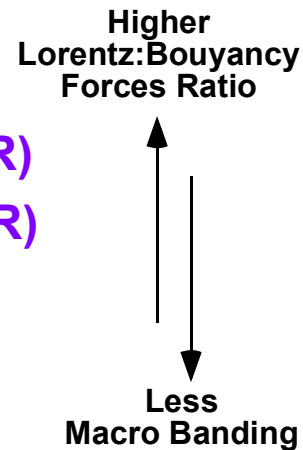
- **Banded**

- Vacuum Arc Remelt (VAR)
- Electro-Slag Remelt (ESR)
- Plasma Arc Melt (PAM)
- Electron Beam (E-beam)

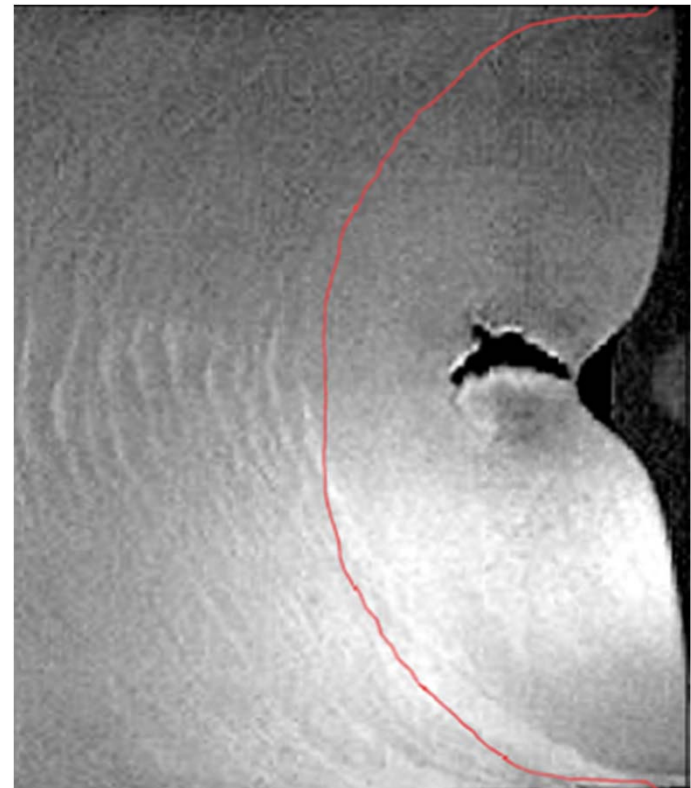
- **No Bands**

(Processes where metal is cast in a mold separate from the melting)

- Vacuum Induction Melted (VIM)
- Skull Melted



VAR Ingot showing bands where power was on during solidification and no bands where hot top solidified with no current

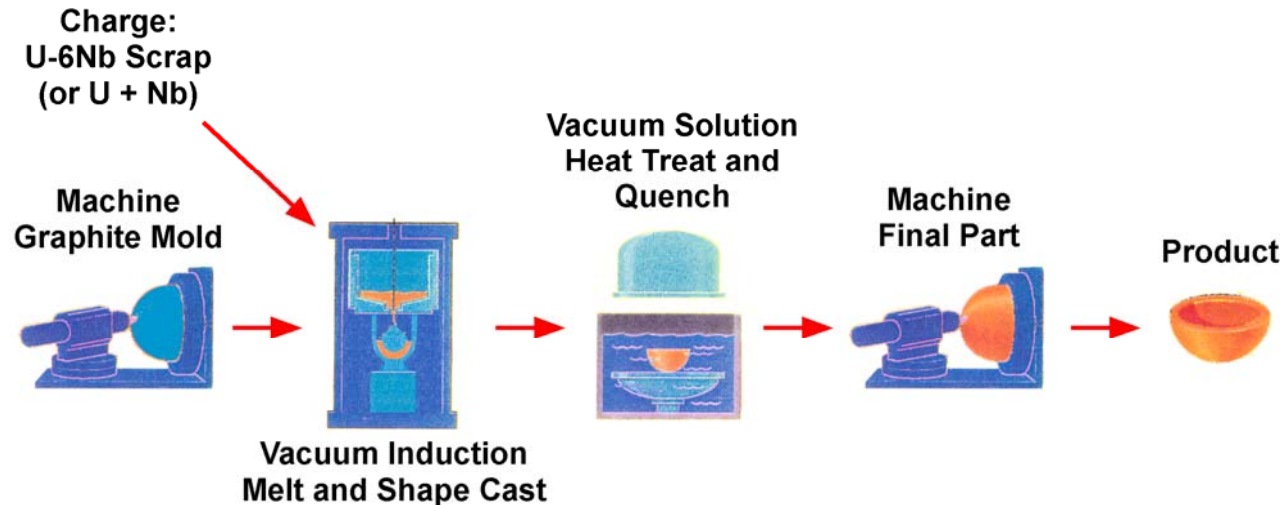


Alternative Processing of U-Nb Alloys

- Direct Cast U-6Nb
- Plasma Arc Melting (PAM)



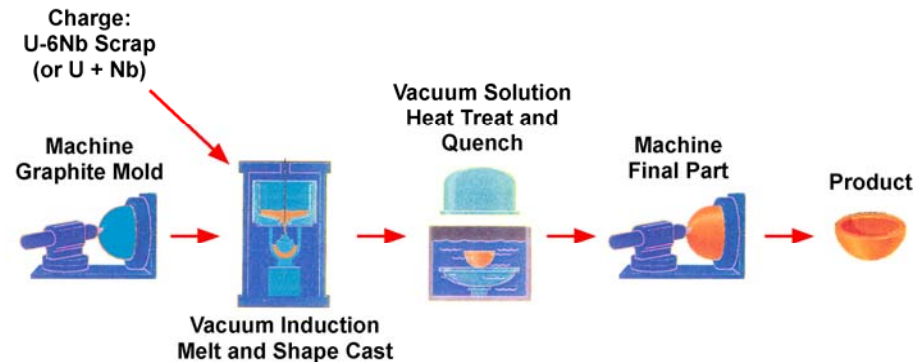
Direct-Cast U-6Nb



Direct-Cast results in:

- Less tooling = Greater manufacturing agility (quicker to first article)
- Less equipment = Reduced facility foot print
- Less equipment = Fewer processes to keep up and running (including maintenance and readiness)
- Fewer processing steps = Lower manufacturing cost
- Fewer processing steps = Lower worker radiologic exposure
- Improved material yield (5% for wrought vs. 40-60% for cast)
- Performance equivalent to wrought

Need for Direct-Cast U-6Nb



Near Term – Short Run Production (hydro components, JTA, ...)

- **Less tooling = Greater manufacturing agility (quicker to first article)**
- **Less equipment = Fewer processes to keep up and running (including maintenance and readiness)**
- **Fewer processing steps = Lower manufacturing cost**

Long Term – All Production in a New Facility

- **Less tooling = Greater manufacturing agility (quicker to first article)**
- **Less equipment = Reduced facility foot print**
- **Less equipment = Fewer processes to keep up and running (including maintenance and readiness)**
- **Fewer processing steps = Lower manufacturing cost**
- **Fewer processing steps = Lower worker radiologic exposure**
- **Improved material yield (5% for wrought vs. 40-60% for cast)**

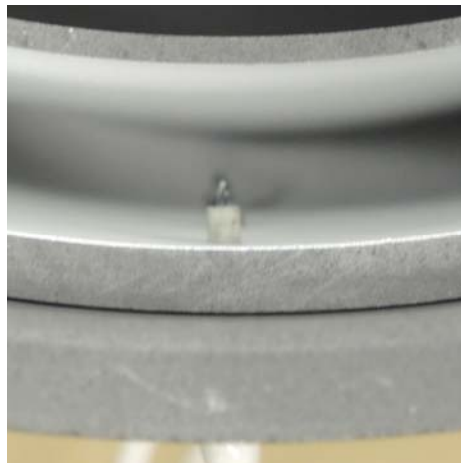
Summary of Direct Cast U-6Nb

- Direct-cast U-6Nb represents an alternative to traditional wrought processing
- To a first approximation, cast has mechanical behavior identical to wrought
 - Microporosity has little effect on quasi-static properties
 - Nearly identical behavior in dynamic expansion
- Full size parts can not be produced in a large single coil furnace
 - Thermal gradient in mold must be above about 500°C/m
 - Counter gravity casting requires a three zone furnace
- Nb segregation is a continuing concern
 - Good casting practice is required
 - Need to solidify quickly or cast with counter gravity solidification
 - For some geometries' casting thickness (degree of near net shape) is limited by distortion during quenching

Instrumented U-6Nb Casting – 250 mm Hemi



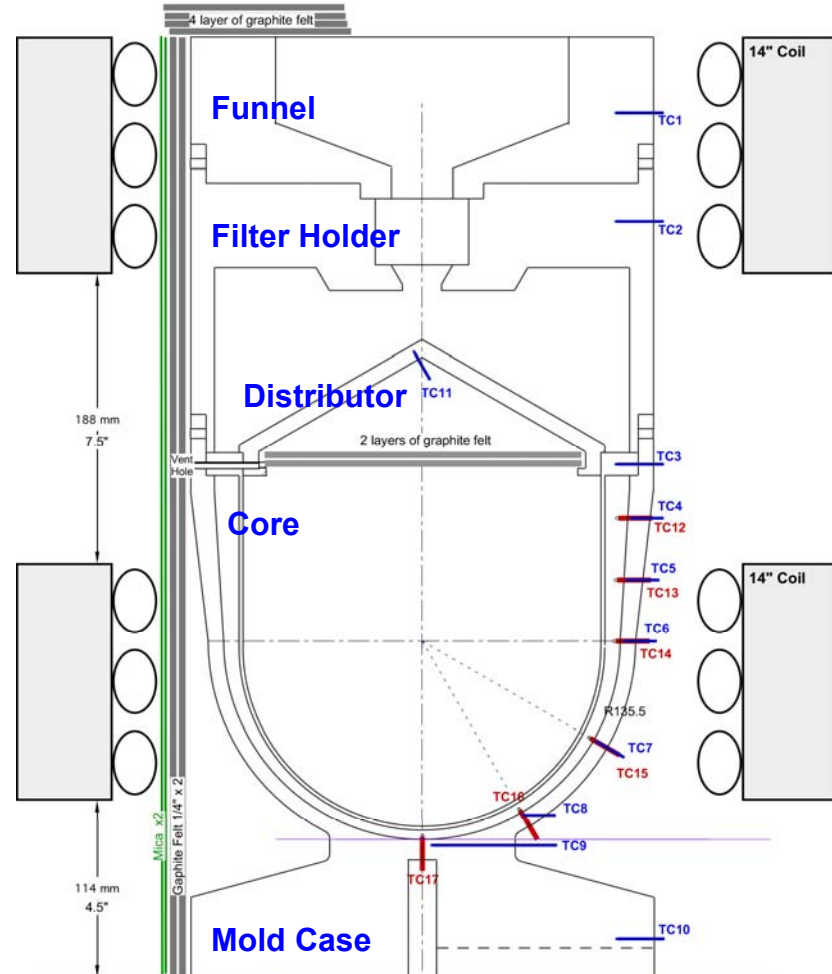
**Bare junction
type-C
thermocouple
in casting
cavity to
measure metal
temperatures**



17K-713

250 MM HEMI
REV F
07/19/16
SCALE 1:2

Funnel - HLM
Filter Holder - HLM
Distributor - HLM
Case - 2020
Core - 2020



Locations of type-K thermocouples in the mold in Blue

Locations of type-C thermocouples in the casting cavity in red

Baseline 17K-713 – Mold Stack and Breakout

**Mold Stack with
Insulation and Coils**



**Mold Stack w/o
Insulation and Coils**



Funnel with Filter

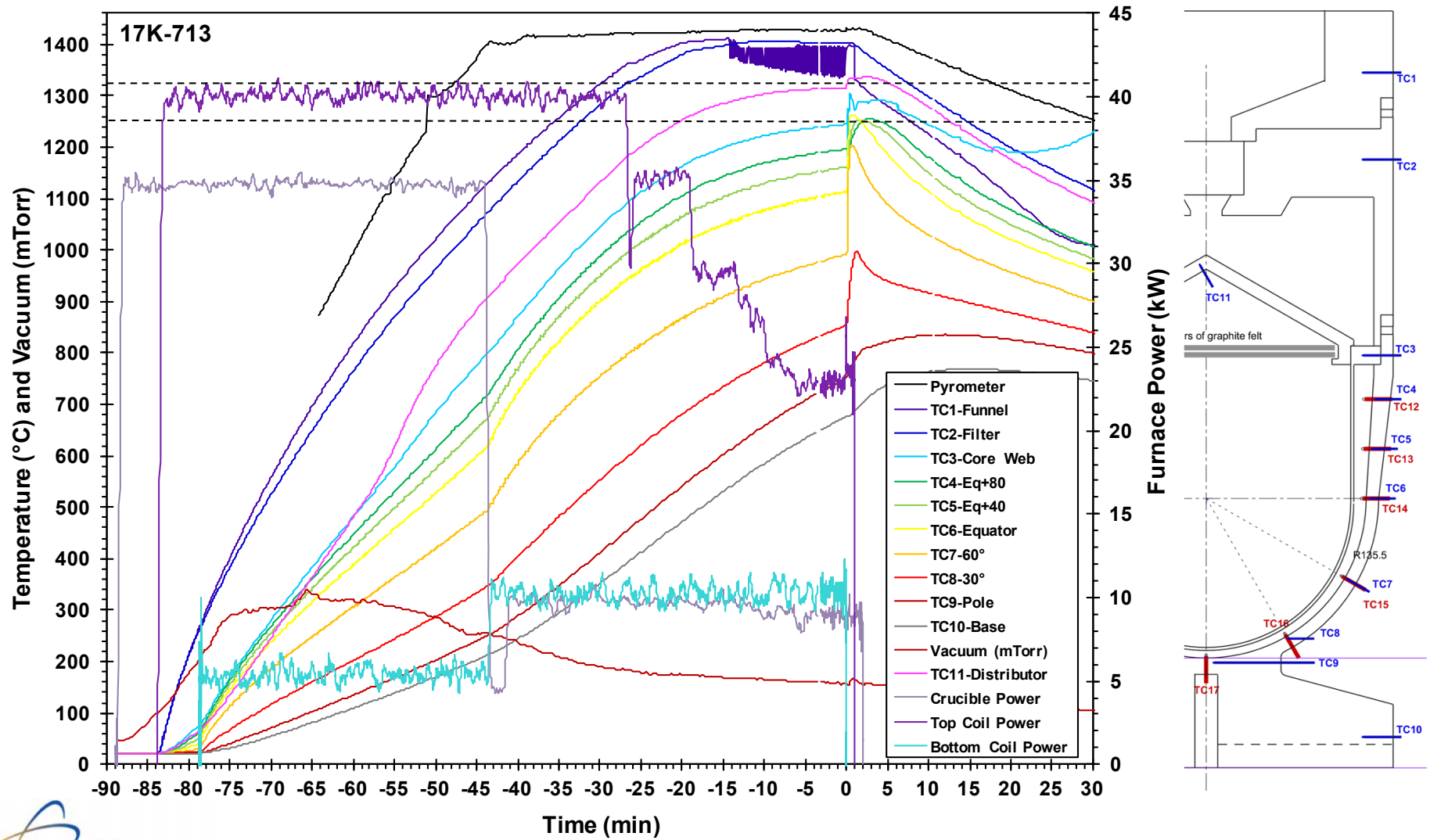


Top of Distributor



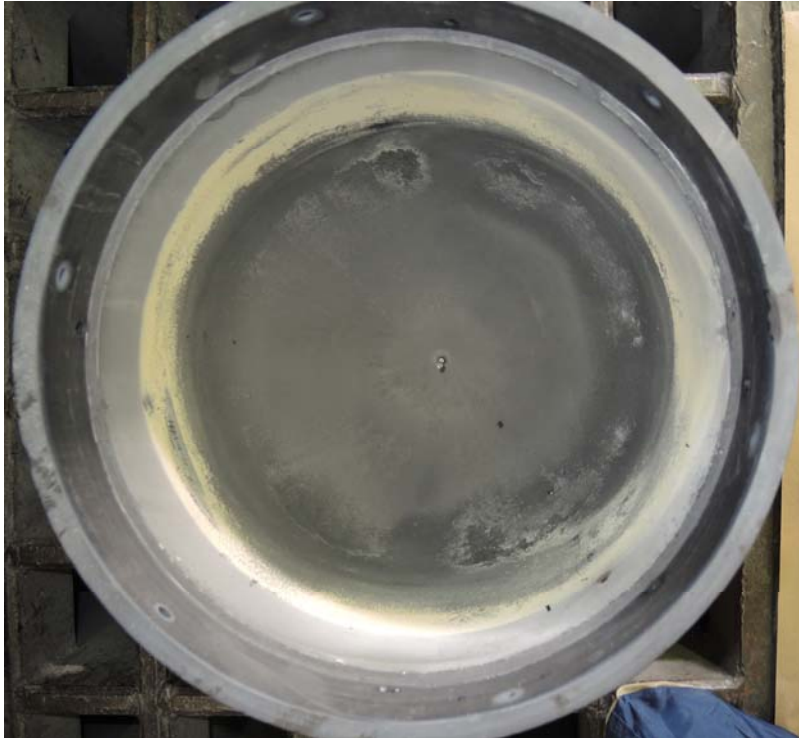
Baseline 17K-713 – Run Chart for 250 mm Hemi

Furnace power settings, vacuum, and temperature of mold as a function of time



Baseline 17K-713 – Breakout

Mold Case with Coating



Raw Casting with Coating



Baseline 17K-713 – Washed Casting

View of Inner Contour

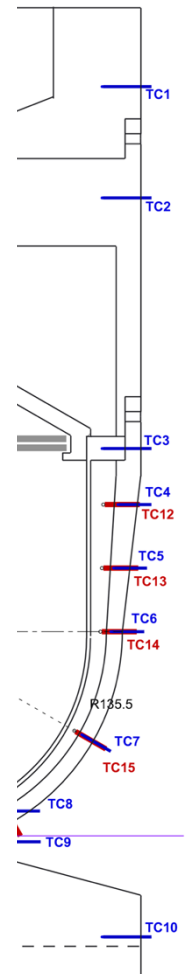
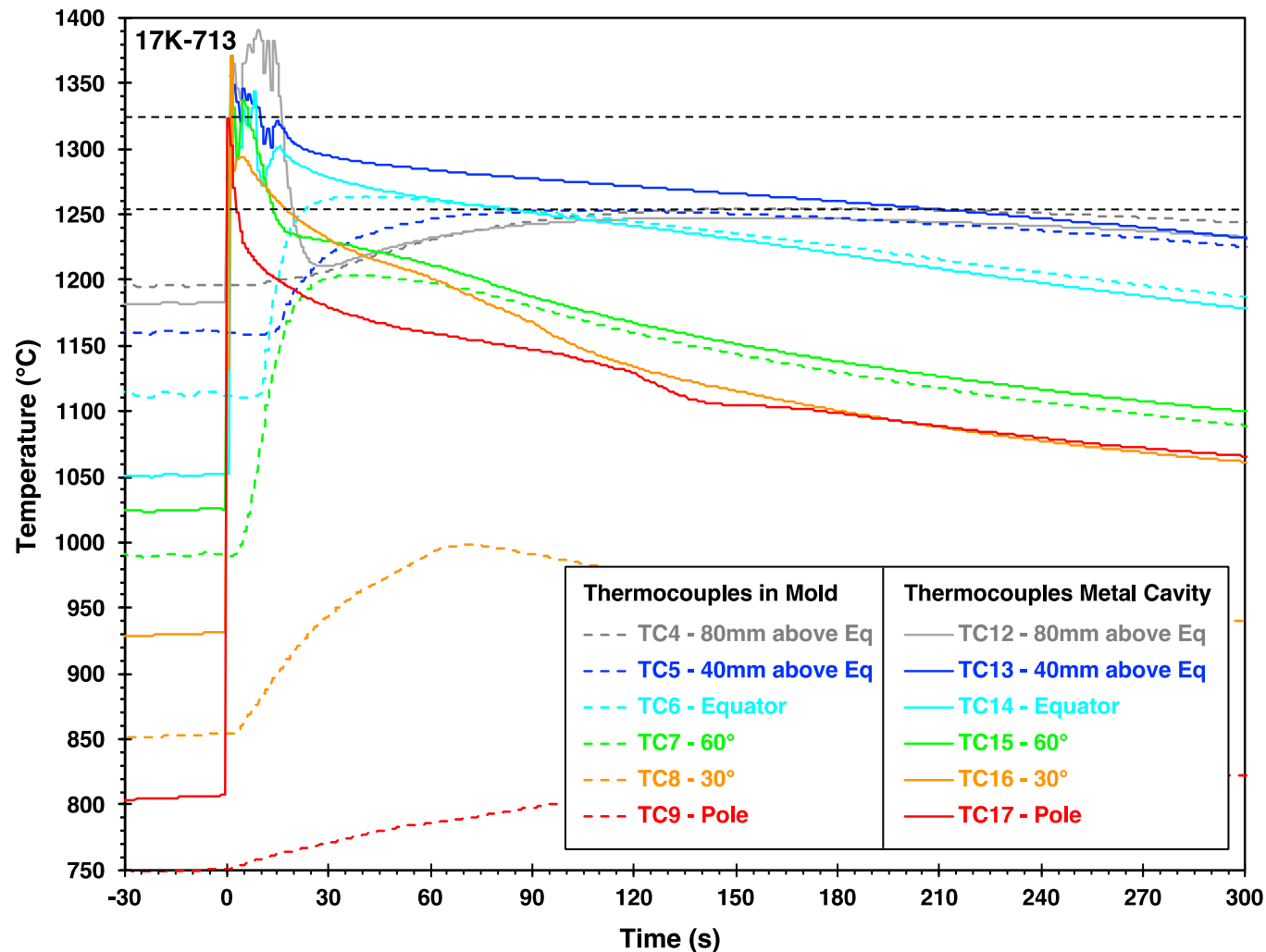


View of Outer Contour



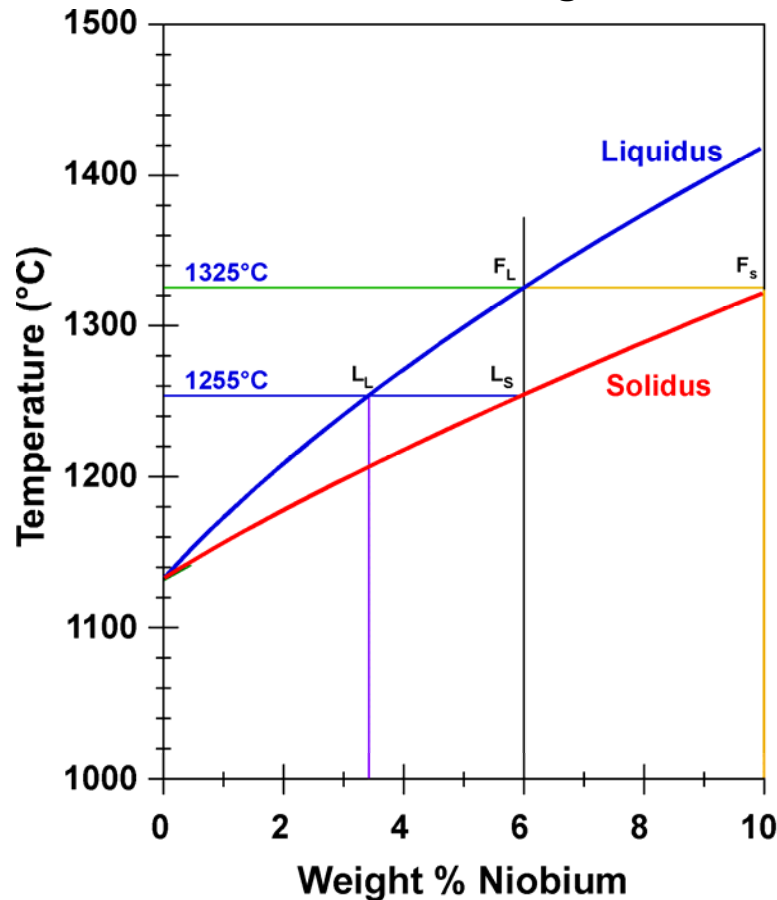
Baseline 17K-713 – Temperature in Casting Cavity

Temperature from thermocouples in casting cavity and mold during solidification

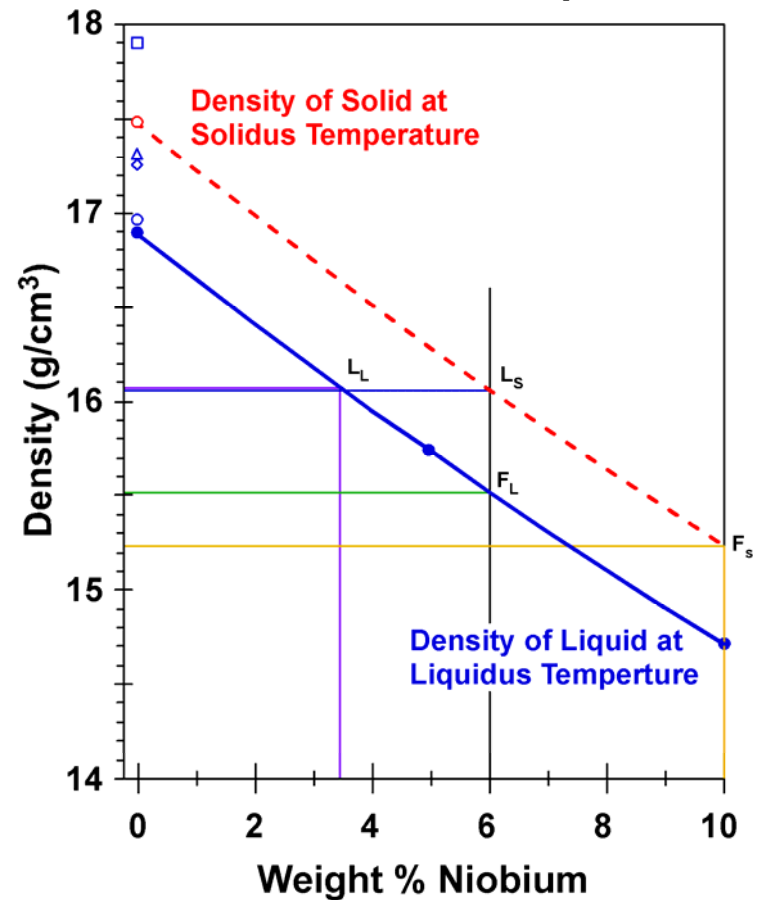


Density Changes During Solidification Make U-6Nb Particularly Prone to Nb Segregation

Uranium Rich Portion of U-Nb Phase Diagram



Density of Solid and Liquid at the Solidus and Liquidus



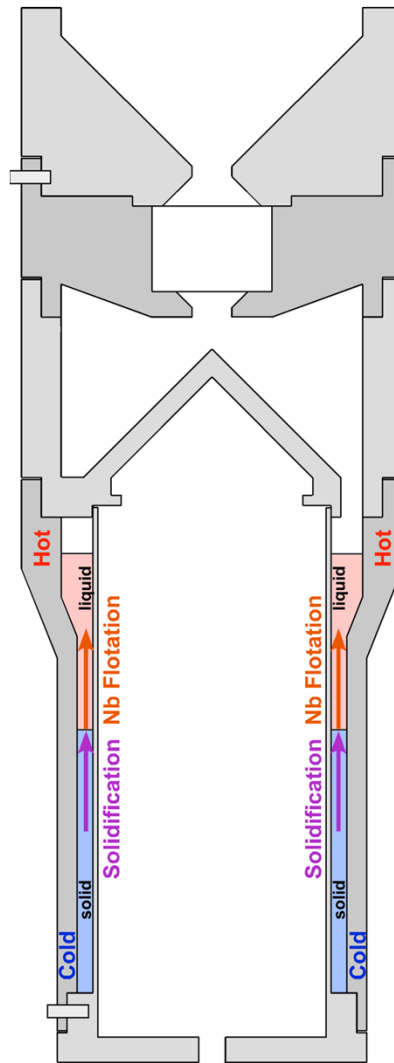
- Liquid density from W.D. Drotning, *High Temp. High Press.*, 14, 253-258 (1982)
- Solid density estimated from published pure U density and Drotning's U-Nb composition dependence of liquid

Growth Direction and Segregation in U-6Nb

Assume Macro-Segregation by Buoyancy Driven Flow

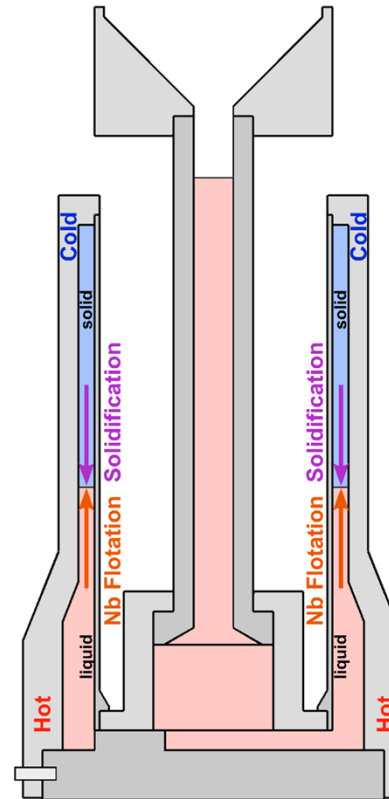
Conventional Mold Design

- Top-to-bottom fill
- Bottom-to-top solidification
- **Solidification front 'chases' Nb flotation**



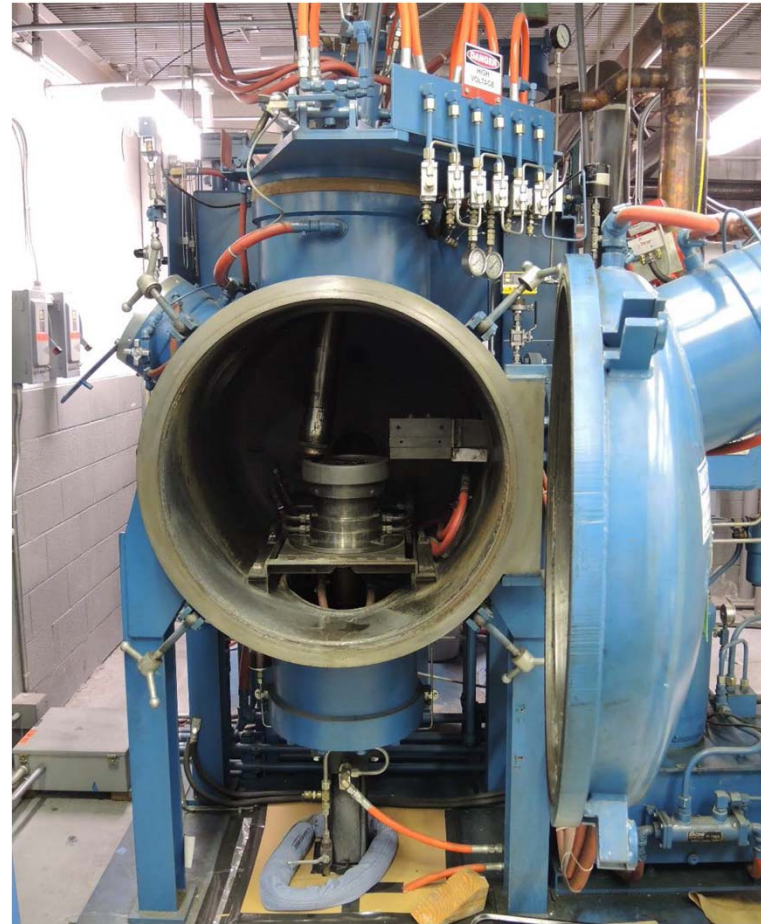
Counter Gravity Solidification Mold Design

- Bottom-to-top fill
- Top-to-bottom solidification
- **Solidification direction is opposite Nb flotation**
- Need to keep down-sprue filled and liquid to maintain pressure on casting and feeder

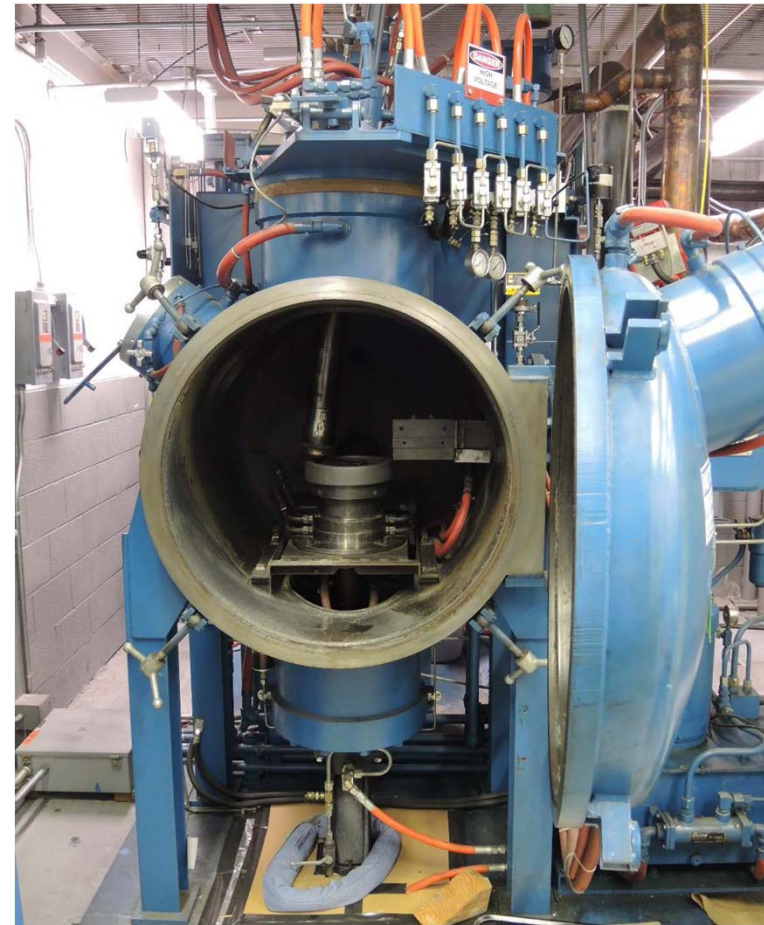
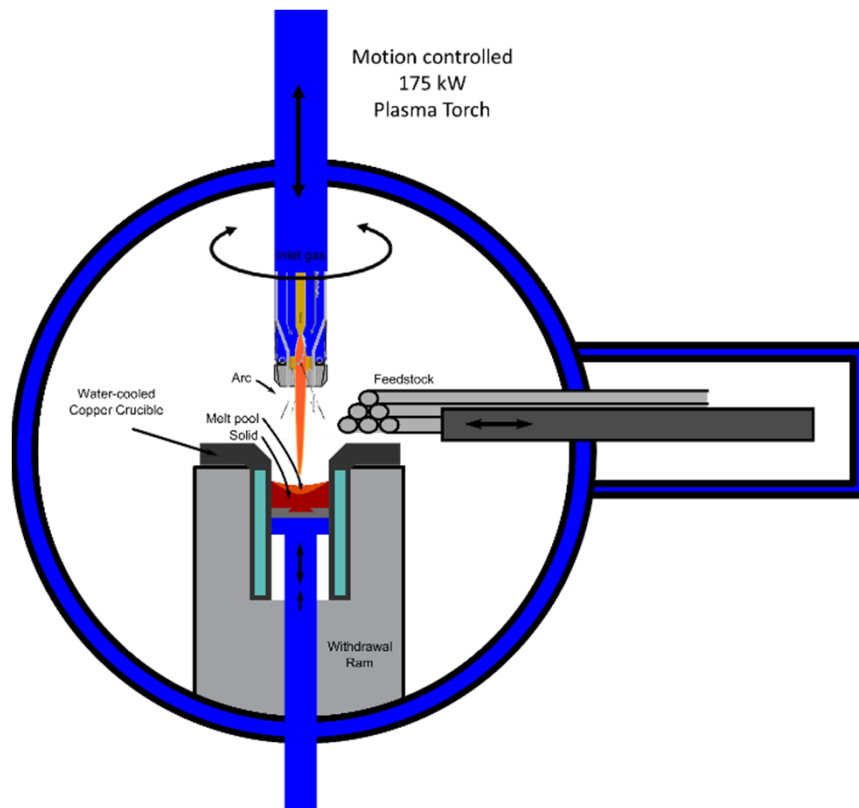


Plasma Arc Melter

- Configured for melting reactive and non-reactive metals
- Designed and build by Retech, installed in 1996
- Single 175 kW plasma gun
- Inert and reactive gas up to 5 psi
- 3" and 4" diameter x 12" crucibles
- Magnetic stirring up to 90 G
- Currently being brought up after many years of being down
- Useful test-bead for potential multi-gun cold-hearth production of DU and DU alloy ingots



Single-Gun Plasma Arc Melter



With Reactive Gas Additions PAM has Potential to Reduce Uranium Oxides and Carbides

